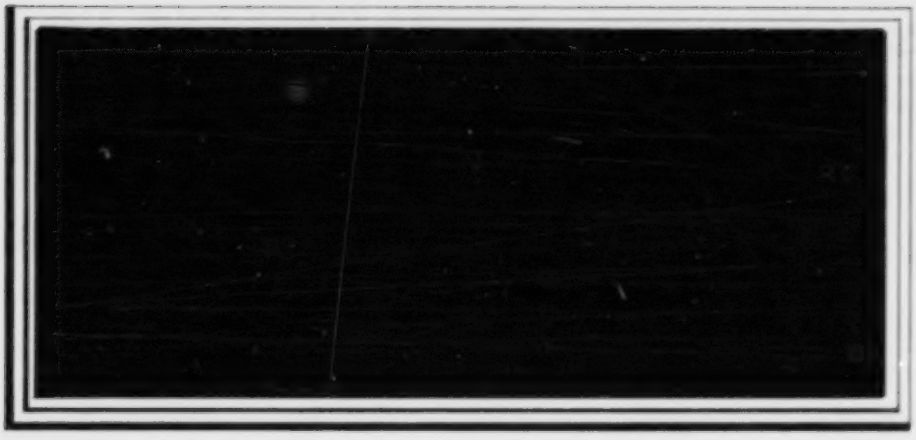
ZENON



90-03963/

INVESTIGATION OF THE USE OF POROUS MEMBRANES FOR THE TREATMENT OF CRUDE OIL EMULSIONS

FINAL REPORT

PREPARED FOR:

DEPARTMENT OF SUPPLY AND SERVICE AND

DEPARTMENT OF ENERGY, MINES & RESOURCES

UNDER CONTRACT NO. 23440-7-9169/01-SQ

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May, 1989

94 pp

MICROMEDIA

CO874074

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EXECUTIVE SUMMARY

The objective of this study was to make an assessment of the technical feasibility of treating water-in-oil emulsions from in-situ bitumen/heavy oil recovery operations using porous membranes. The objective of treatment is to remove the water from the oil to meet product quality specification. Pipeline specifications for water in oil are 0.5% water.

In this study, the feasibility of two approaches to treatment of water-in-oil emulsions using porous membranes were evaluated. In the oil permeation process oil is driven through the membrane by the applied pressure and the oleophilic nature of the membrane material while the water in the emulsion is retained and concentrated on the feed side of the membrane because of the size of the water droplets and the hydrophobic nature of the membrane material.

The other process evaluated was a water permeation process. The objective of this proposed process was to destabilize the water-in-oil emulsion by removal of the salts and surfactants which stabilize the emulsion. In this process water and caustic are added to the water-in-oil emulsion to produce an oil-in-water emulsion and then the water together with the extracted salts and surfactant is removed by porous membrane filtration. By this process a clean water-free oil may be obtained.

This study has shown that ultrafiltraiton of water-in-crude oil emulsions can reduce water concentrations in the oil

permeate from up to 20% water to less than 0.5% water. Unfortunately, due to the high viscosities of water-in-crude oil emulsions the flux rates are so low that the process is not technically feasible.

Ultrafiltration of oil-in-water emulsions formed by the addition of water and caustic to a water-in-crude oil emulsion was shown an ineffective means of extracting the surfactants and salts and producing a clean water-free oil. However, testing has proven that ultrafiltration membranes can maintain a high flux while effectively removing water from an oil-in-water emulsion containing very high concentrations of oil (up to 60%).

1.0 INTRODUCTION

This report presents the results of a project contracted to ZENON Environmental Inc. by the Department of Supply and Services Canada on behalf of Energy, Mines and Resources Canada.

The objective of this study was to make an assessment of the technical feasibility of treating water-in-oil emulsions from in-situ bitumen/heavy oil recovery operations using porous membranes. The objective of treatment is to remove the water from the oil to meet product quality specification. Pipeline specifications for water in oil are 0.5% water.

Water is present in large quantities in the wellhead oil pumped from in-situ operations for the recovery of heavy oil and bitumen. This water is emulsified with the crude oil and is described as water-in-oil emulsions. Other crude oil treatments introduce water into the oil such as in crude-oil desalting. In all of these cases, the water must be removed to meet product quality specifications or to reduce corrosion and foaming difficulties during refinery operations.

The conventional methods of water removal have been successful for conventional crudes. However, the use of in-situ methods for the recovery of heavy oil and bitumen have caused the formation of stable water-in-oil emulsions that resist conventional treatment and chemicals or coalescent media. In particular, the fireflood method of in-situ recovery creates water-in-oil emulsions that are extremely stable. Even if water is removed by azeotropic distillation, the remaining salts cause

problems. Present practice in oil production permits the blending of wet crudes with suitable dry crudes to pass pipeline specifications for bottom sediments and water (BS&W).

The nature of separations with porous membranes from other systems have shown that membranes can selectively remove various components from solution. This has been particularly successful for low oil-in-water emulsions such as oil field produced water.

In this study, the feasibility of two approaches to treatment of water-in-oil emulsions using porous membranes were evaluated. In the oil permeation process oil is driven through the membrane by the applied pressure and the oleophilic nature of the membrane material while the water in the emulsion is retained and concentrated on the feed side of the membrane because of the size of the water droplets and the hydrophobic nature of the membrane material. The other process evaluated was a water permeation process. The objective of this proposed process was to destabilize the water-in-oil emulsion by removal of the salts and surfactants which stabilize the emulsion. In this process water and caustic are added to the water-in-oil emulsion to produce an oil-in-water emulsion and then the water together with the extracted salts and surfactant is removed by porous membrane filtration. By this process a clean water-free oil may be obtained.

2.0 CONCLUSIONS AND RECOMMENDATIONS

The following is a summary of the most important conclusions made based on the results of this study.

1. Ultrafiltration of water-in-crude oil emulsions can reduce water concentrations in the oil permeate from up to 20 percent water to less than 0.5 percent water. Unfortunately, due to the high viscosities of water-in-crude oil emulsions the flux rates are so low that the process is not technically feasible.

2. Ultrafiltration of oil-in-water emulsions formed by the addition of water and caustic to a water-in-crude oil emulsion is not an effective means of extracting the surfactants and salts and producing a clean water-free oil. However, testing has confirmed that ultrafiltration membranes can maintain a high flux while effectively removing water from an oil-in-water emulsion containing oil in concentrations up to 60%.

Although the two processes assessed were not technically feasible for the water-in-oil emulsions, the process may have applicability for the treatment of other streams. The oil permeation studies proved that oleophilic/hydrophobic membranes can be used to produce a clean water-free oil permeate. This process may be applicable for the production of specialty oils that are produced in relatively low quantities. The water permeation studies proved that water can be extracted from a stable oil-in-water emulsion even at very

high oil concentrations (up to 60% oil). These results may be applicable to on-gong studies of ultrafiltration of produced water emulsions.

3.0 EXPERIMENTAL PROCESS DEVELOPMENT

3.1 Overall Experimental Strategy

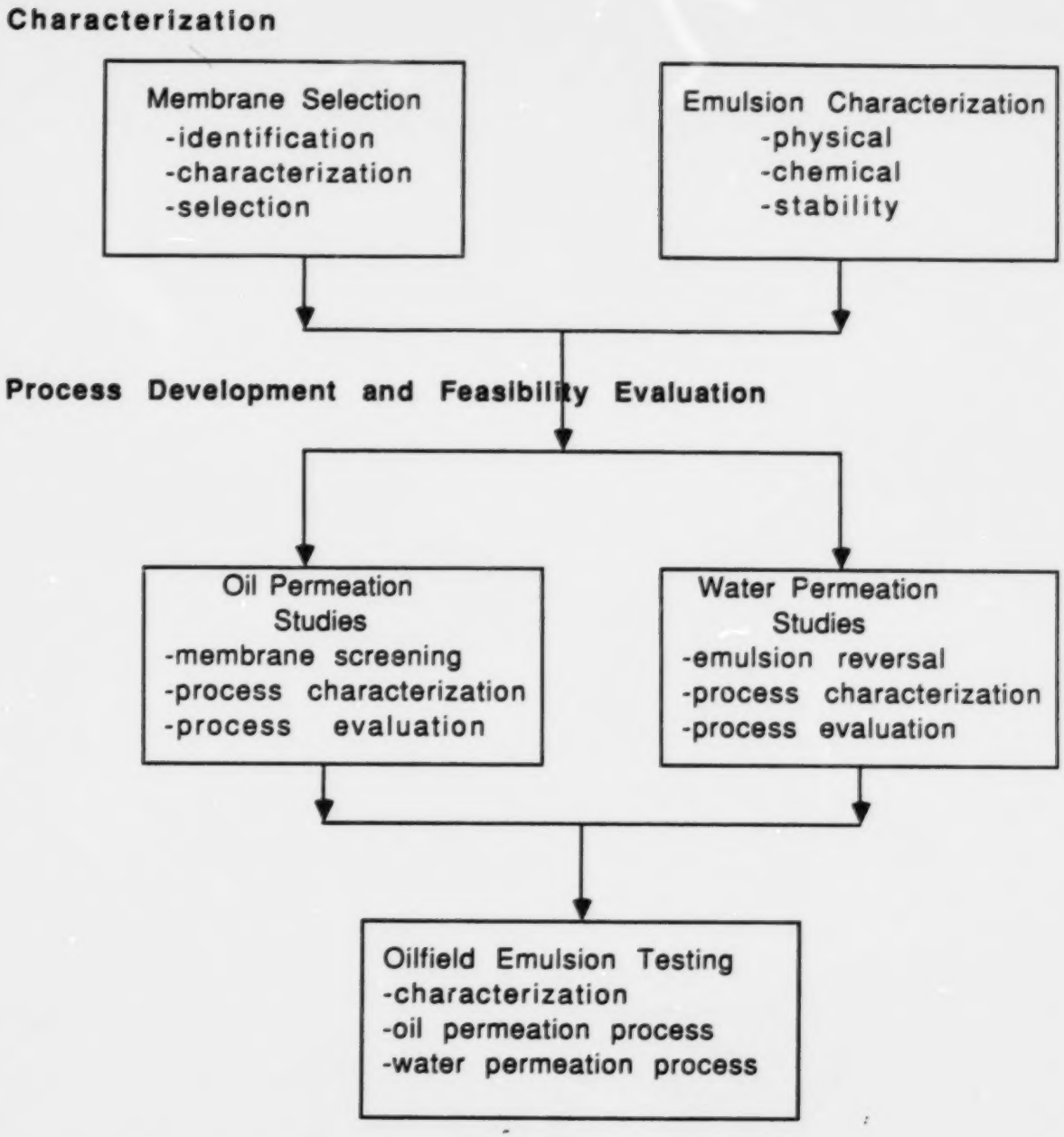
The experimental program was designed to provide information regarding the technical feasibility of using porous membranes for the treatment of water-in-oil emulsions. The experimental work program is illustrated schematically in Figure 3-1. Preliminary testing included characterization of a sample of water-in-oil emulsion obtained from an oil producer in western Canada and identification and characterization of suitable porous membranes. Process development focused on two distinctly different processes. The first process investigated was removal of the contaminants by permeation of the oil through the porous membrane to the exclusion of the water, sediments and salts. The second process investigated involved creating an oil-in-water emulsion from the water-in-oil emulsion then causing the water and salts to permeate while excluding the oil. In final testing the applicability of these two processes were evaluated for three different oilfield emulsions.

3.2 Emulsion Characterization

A 200 L sample of water-in-oil emulsion for process development was supplied by Shell Canada. This wellhead emulsion was taken at the Peace River, Alberta site in the spring of 1988. For testing process applicability, three 20 L water-in-oil samples were supplied by the Alberta Research Council sample bank. The samples were from Cold Lake, Peace River and Lloydminister.

All samples were characterized with respect to physical and chemical parameters including water content, sediments, metals,

Figure 3.1 Overall Experimental Strategy



viscosity and droplet size. Details of the analytical methods used for these characterizations are included in Appendix A.

The stability of each of these emulsions with respect to time and temperature up to 80°C was also examined.

3.3 Membrane Selection and Characterization

Initially a detailed list of available membrane chemistries, and details of film supplier, trade name, membrane suppliers and available configurations was complied. This list was compiled based on the published literature, manufacturer contacts and trade literature.

Samples of films of polymers from which potentially suitable membranes are made were obtained. The contact angle with water for each of the films was determined at ambient conditions of temperature, pressure and humidity. The contact angle measurement was made using the tilting plate method. The general arrangement is indicated schematically in Figure 3-2. A plate of the solid dips into the liquid and its position is adjusted until the liquid surface is perfectly flat right up to the surface of the solid. The angle between the liquid surface and the solid surface is the contact angle. Details of the method used to measure contact angle in this study are included in Appendix A.

The tilting plate method is generally considered to be the most accurate method of measuring contact angle. However, this contact angle measurement is sensitive to the condition of the solid surface (roughness, cleanliness etc). For this reason contact angles cannot be measured for porous membranes however, contact angles can be measured

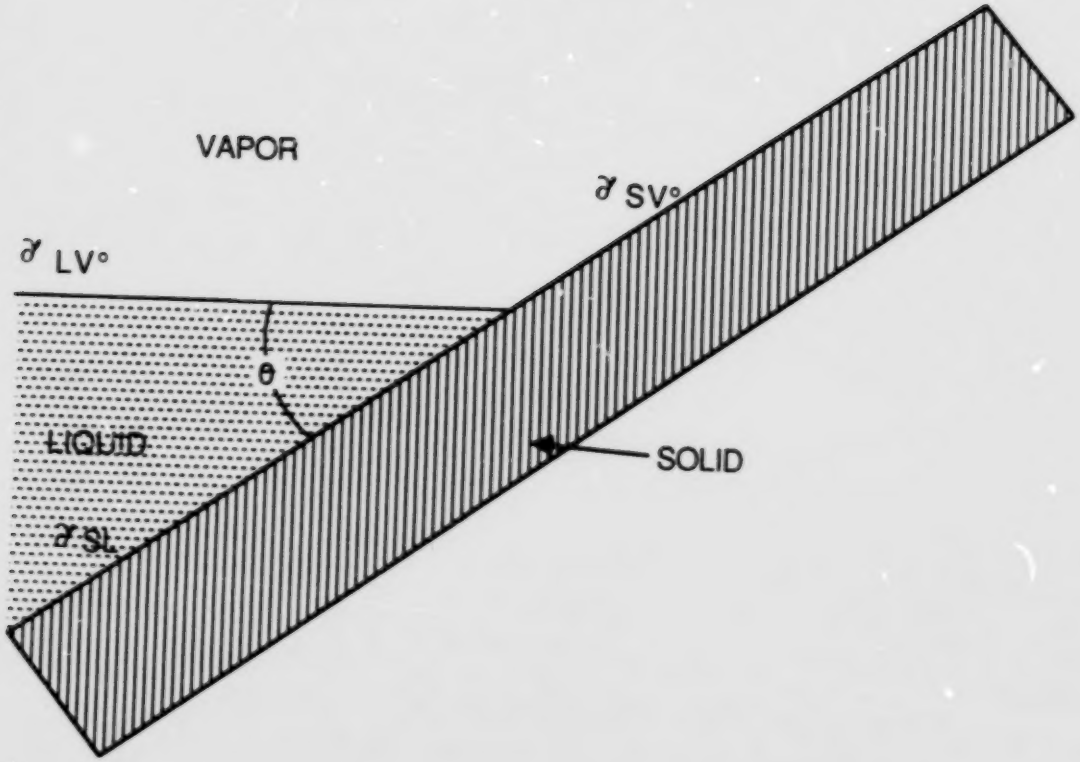


FIG 3.2 THE TILTING PLATE METHOD FOR CONTACT ANGLE MEASUREMENT

for films of the material from which each membrane is made.

3.4 Bench-Scale Process Equipment

The equipment used in the bench scale testing consisted of a 25 liter feed tank, a heating and cooling system, a high pressure pump, the necessary valves and piping, flowmeter, pressure gauges and membrane cell. The entire system was constructed from stainless steel. A schematic of this layout can be seen in Figure 3-3.

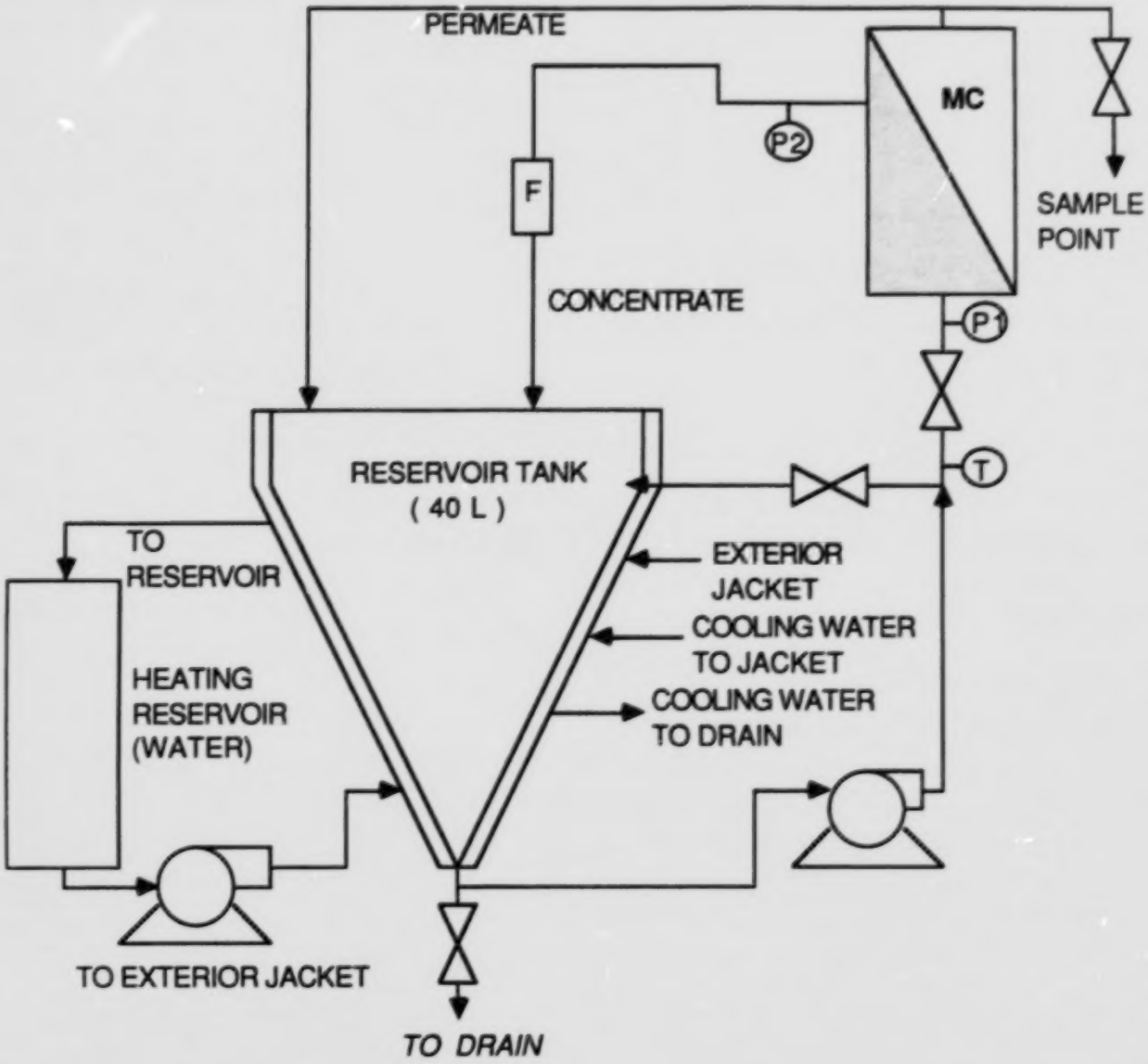
The equipment was operated in the following manner: The pump received the emulsion through an intake in the bottom of the feed tank. From the pump a portion of the feed was fed to the membrane cell and the rest is recirculated into the tank. The pressure in the membrane cell was regulated by intake and outlet valves. After passing through the membrane, the concentrate was returned to the feed tank and the permeate could be returned or collected.

Two test membranes configuration were used during this study and a detailed description of both can be found in the Appendix A. The two types of configuration used were half inch tubular and flat sheet. The tubular configuration was the configuration of choice if both configurations were available because it has superior flow characteristics and larger surface area.

3.5 Oil Permeation Development Tests

The process tested in the oil permeation development tests is illustrated in Figure 3-4. Oil is driven through the membranes by the applied pressure and the oleophilic nature of the membrane material while the water in the emulsion is retained and concentrated on the

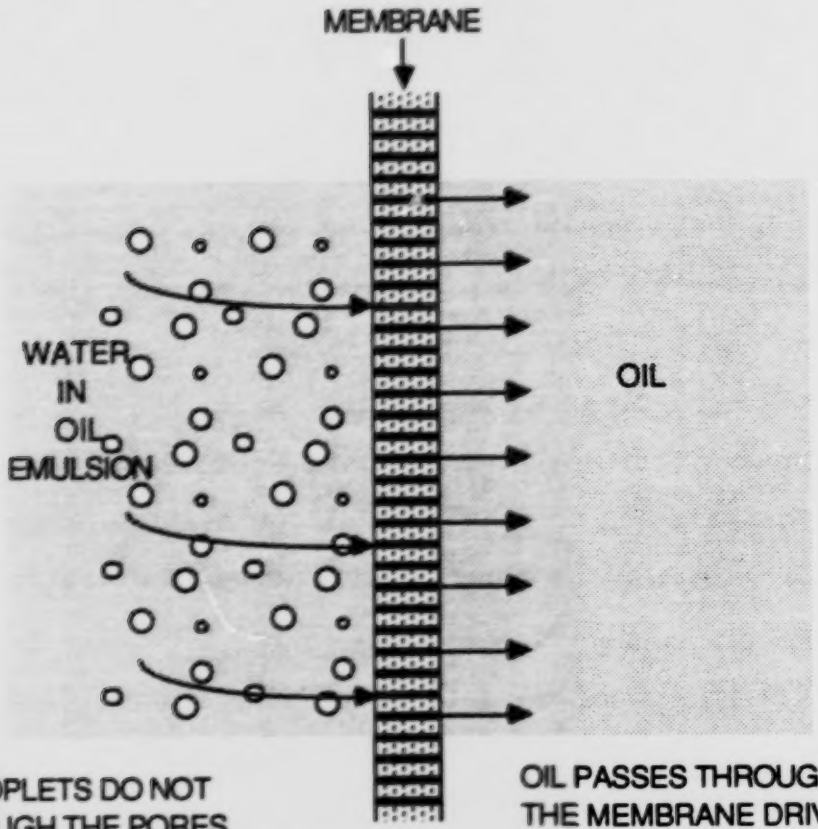
FIGURE 3.3 **SCHEMATIC DRAWING OF BENCH-SCALE EMR TESTING SYSTEM**



LEGEND :
MC = membrane cell
P1 = inlet pressure gauge
P2 = outlet pressure gauge
T = temperature gauge
F = flow gauge

(note : the hold -up volume of the system is 2.2 L)

Figure 3.4 OIL PERMEATION PROCESS



WATER DROPLETS DO NOT PASS THROUGH THE PORES DUE TO THE SIZE OF THE DROPLETS AND THE HYDROPHOBIC NATURE OF THE MEMBRANE MATERIAL

OIL PASSES THROUGH THE PORES OF THE MEMBRANE DRIVEN BY THE APPLIED PRESSURE AND THE OLEOPHILIC NATURE OF THE MEMBRANE MATERIAL

feed side of the membrane due to exclusion from the membrane pores because of the size of the water droplets and the hydrophobic nature of the membrane material.

In initial tests, six membrane material types were assessed. The membranes to be tested were selected based on the results of the membrane characterization and selection. Table 3-1 shows the membranes used and the test conditions. All membranes used were conditioned by soaking in mineral spirits for 24 hours prior to use. The performance parameters measured were the membrane permeation rate and the water content of the feed and permeate.

Based on the results of the initial tests, the most promising membrane was selected. This membrane was used in a series of tests conducted to characterize and optimize the oil permeation process. The permeate rate and permeate water content were determined over a range of operating pressures, operating temperatures and flowrates as shown in Table 3-1. Based on these tests, the optimum operating conditions were determined.

3.6 Water Permeation Development Tests

The objective of the water permeation development tests was to evaluate the potential of dialysis treatment for salt and surfactant removal and destabilization of the water in oil emulsion.

In conventional desalting and dewatering, water and caustic is mixed with the crude oil emulsion. The resulting emulsion is then destabilized in an electric field (Barnett, 1988). The dialysis treatment tested in this study is analogous. Water and caustic are added to the emulsion with controlled mixing until a stable emulsion is

Table 3-1 OIL PERMEATION DEVELOPMENT TESTS

Membrane Screening Tests

Membrane Type	Pore Size		Configuration	Operating Temperature (°C)	Operating Pressures (psi)
Polyether Sulphone	10K	40K	tubular	60 , 75	50 , 100
Hydrophilic Polyaromatic	10K	50K	tubular	60 , 75	50 , 100
Polysulphone	10K	50K	tubular	60 , 75	50 , 100
Polypropylene	0.02µ (30K)	0.1µ (200K)	flat sheet	60 , 75	30 -> 100
Teflon	0.1µ (200K)	1.0µ	flat sheet	60 , 75	30 -> 100
Ceramic	1.0µ		tubular	60 , 80	90

Process Characterization Tests

Teflon:	
Pore Size	0.5µ, 1.0µ
Configuration	flat sheet
Temperature	40°C to 60°C
Inlet Pressure	40 to 95 psi
Feed Flowrate	7 to 9 L/min

formed. The emulsion is then treated by ultrafiltration in an effort to remove the added water and the salts and surfactants which stabilized the water in oil emulsions.

The first step in testing was to determine the conditions necessary to form a stable oil in water emulsion from the water in oil emulsion.

Initially the stability of a one to one mixture of water and water-in-oil emulsion was tested at several pH's and temperature. The pH was adjusted using a solution of NaOH as appropriate. Following this test, a titration was performed on a one to one mixture of emulsion and water to determine the amount of hydroxide that must be added to reverse the emulsion. An accurate pH reading could not be made so readings were taken on the permeate when it was processed. From this data it was possible to determine the amount of NaOH that was needed to make a stable oil-in-water emulsion for ultrafiltration.

The membranes used in this study were selected based on results of an on-going study (Produced Water Ultrafiltration EOR (Enhanced Oil Recovery) Field Tests) performed at ZENON Environmental. In that study, extensive testing was performed on three membranes and it was found that the permeate quality was only acceptable in two of them, HST and HEM. Along with the two tested membranes a standard wastewater membrane was tested in our application (HSC). The test system used is the system described in Section 3.4.

The testing was performed to determine the quality of the permeate and the emulsion after processing and the flux rates. Pure water flux rates were taken on the membranes to check the baseline flux. The conditions which the system was run at were 20 L/min, inlet

pressure 70 psi, outlet pressure 55 psi and a temperature of 40°C.

The first dialysis tests involved a one to three mixture of emulsion and water. During this trial the permeate was continuously removed and the concentrate returned to the feed reservoir. By this process the water and associated salt and surfactants were removed and the oil concentrated in the feed reservoir. The trial was continued for 2 hours.

Following this test, a stabilization test was performed on the concentrate which was produced in the first test. For this run the concentrate was again diluted with 10 L of tap water and run for 4 hours while recirculating the permeate and the concentrate back into the feed tank.

3.7 Oilfield Emulsion Tests

In order to investigate the potential application of the oil permeation process and the dialysis water permeation process for treatment of oilfield water-in-crude oil emulsions, three emulsion samples were obtained and tested.

Oil permeation tests were carried out for each of the three emulsion samples. The test equipment used was the same equipment described in Section 3.4. For each test the membrane was the Teflon 1.0 um flat sheet, each test was run at 40°C and 90 psi applied pressure. A new membrane which had been soaked in mineral spirits for 24 hours was used for each trial.

The very high viscosity of the emulsions limited the flow through the membrane cell thus each test was run at the maximum flow possible. Each trial was run for 5 hours or until the permeate rate

became too low to measure. The parameters measured during each trial were the permeate rate, the water content in the permeate and the water content in the feed.

Dialysis water permeation tests were attempted for each of the three emulsions. For each emulsion the conditions necessary to form a stable oil in water emulsion were determined. The stability of a one to one mixture of water and water in oil emulsion was tested at several pH's. The pH was adjusted using a sodium hydroxide or sulphuric acid as appropriate.

A water permeation trial was performed to determine the quality of the permeate and the emulsion after processing and the flux rates. The membranes used were the HST, HEM and HSC half inch tubes. The conditions which the system was run were 20L/min, inlet pressure 70 psi, and a temperature of 40°C. During the trial the permeate was continuously removed while the concentrate was returned to the feed reservoir. Following this a stability test was performed on the concentrate. The concentrate was re-diluted with 10 L of tap water and run for 2 hours while recirculating the permeate and the concentrate back into the feed tank.

4.0 RESULTS AND DISCUSSIONS

4.1 Emulsion Characterization

Over the course of this study, four water-in-oil emulsions were received and characterized. Upon receipt each of the wellhead emulsion samples were visually examined. The Peace River 1 wellhead emulsion obtained in the spring of 1988 appeared to be homogeneous, viscous, black oil emulsion. The sample had a strong odour of hydrogen sulfide. The Peace River 2 wellhead emulsion obtained in the fall, 1988 was not homogeneous as received. The sample had brown water floating on top of the viscous black oil emulsion. The water layer was discarded before characterization and testing. The oil layer was very viscous and smelled of hydrogen sulphide. The Cold Lake wellhead emulsion sample was black, homogeneous and very viscous while the Lloydminister sample was black, homogeneous and less viscous than the other samples. All samples appeared to be stable over the test period.

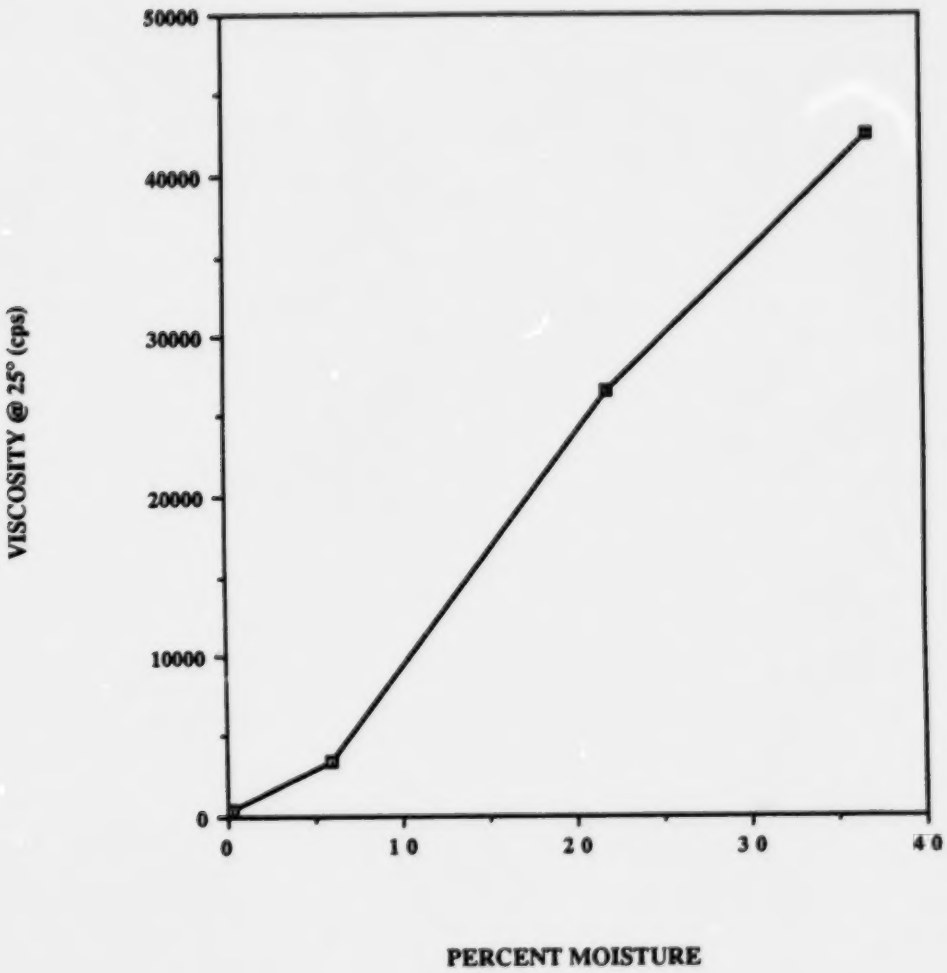
Table 4-1 shows the results of the chemical analysis of these samples. The moisture content of these samples range from 0.25% for the Lloydminister sample to 37% for the Peace River 2. The Lloydminister sample is acceptable with respect to water content for pipelining without further treatment.

The viscosity was also observed to vary widely between samples. Lloydminister viscosity was lowest at 50 cps while Peace River 2 was highest at 42,500 cps. The viscosity was observed to vary directly with water content. Figure 4-1 shows the very clear relationship between viscosity and water content for the four emulsions studied. For every percent increase in moisture the viscosity increases approximately 1100 cps.

Table 4.1 Water in Oil Characterization

Parameter (mg/l)	Peace River 1 spring 1988	Peace River 2 fall 1988	Lloydminster fall 1988	Cold Lake fall 1988
Moisture (%) - KFT	6.0	37	0.25	22
Viscosity @ 24°C	3420 cps	42500	500	26500
Specific Gravity (60/60 ° F)	0.976			
Sediments (%) (Toluene Extration)	0.040	0.15	0.45	0.23
Calcium	58	110	63	150
Magnesium	< 20	< 20	< 20	< 20
Sodium	420	920	140	760
Potassium	< 100	< 200	< 200	200
Aluminum	< 100	43	15	40
Barium	1	1.8	1.2	1.6
Beryllium	< 1	< 200	< 200	< 200
Boron	< 200	58	16	74
Cadmium	< 0.5	< 0.5	< 0.5	< 0.5
Chromium	6.9	0.66	0.75	2
Cobalt	< 3	< 1	< 1	< 1
Copper	< 2	< 1	< 1	< 1
Iron	28	63	15	20
Lead	< 10	< 5	< 5	< 5
Manganese	1	< 2	< 2	< 2
Molybdenum	10	< 1	< 1	< 1
Nickel	59	54	45	57
Phosphorus	47	32	< 30	< 30
Silicon	< 200	55	< 20	54
Silver	< 3	< 3	< 3	< 3
Strontium	0.9	3.2	0.32	0.34
Sulphur	43000	42000	28000	33000
Thallium	< 20	< 1	< 1	< 1
Titanium	6.6	12	9.5	14
Vanadium	170	160	94	150
Zinc	2	4.2	6	7.7
Zirconium	< 3	< 3	< 3	< 3

Figure 4.1 VISCOSITY VS PERCENT MOISTURE



The viscosity of water in oil emulsion is also dependent on temperature. Figure 4-2 shows the viscosity as a function of temperature for the four emulsions examined. This figure shows the strong dependence of viscosity and temperature for these emulsion.

Viscosity is an important factor in the mass transfer at the membrane surface. The effect of viscosity on the membrane processing of these water-in-oil emulsions will be discussed in later sections.

Also included in Table 4-1 is the concentrations of metals in each of the emulsion samples. The observed metals concentration are within the range expected for these types of emulsions. The high sulphur concentrations are typical of the heavier oils found in Alberta and exists in may forms including hydrogen sulphide. Overall, the Lloydminister sample has the lowest metals content.

Figure 4-3 shows the droplet size distribution for the Peace River 1, Peace River 2 and Cold Lake water-in-oil emulsions. Lloydminister is not included because of its low water content. The limitation of this analysis is that the minimum droplet diameter measurable is 0.7um for these samples. An examination of the droplet size distribution for each of these emulsions indicates that a considerable fraction of the droplets would be less than 0.7 micrometers. This suggests that for membrane processing the pore size would have to be considerably smaller than 0.7 micrometers to exclude the water droplets.

The stability of each of the emulsion samples with respect to temperature up to 80°C for up to 4 hours was evaluated. In all cases, the emulsion did not destablize with heating or subsequent cooling.

Figure 4.2 VISCOSITY PROFILE FOR WATER IN OIL EMULSIONS

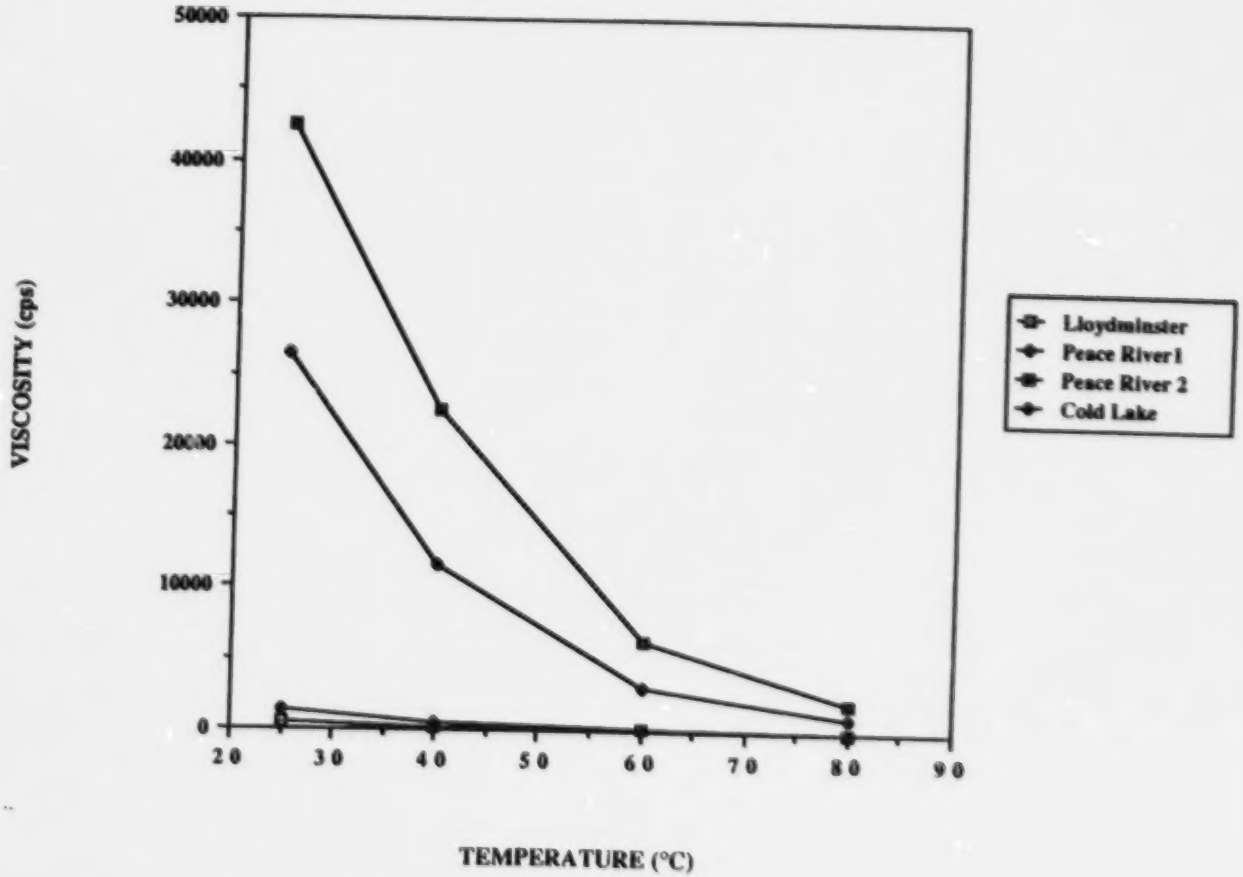
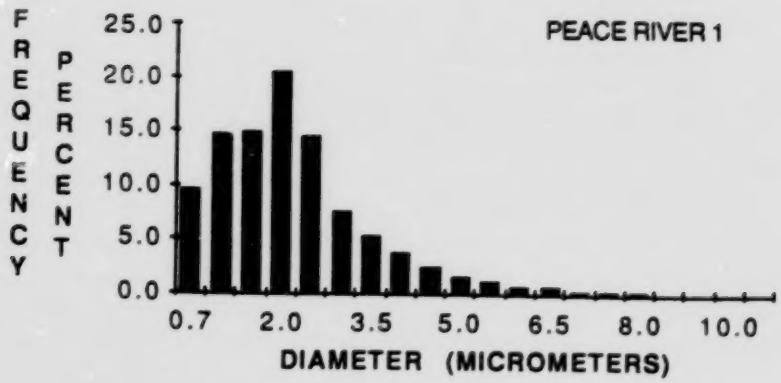
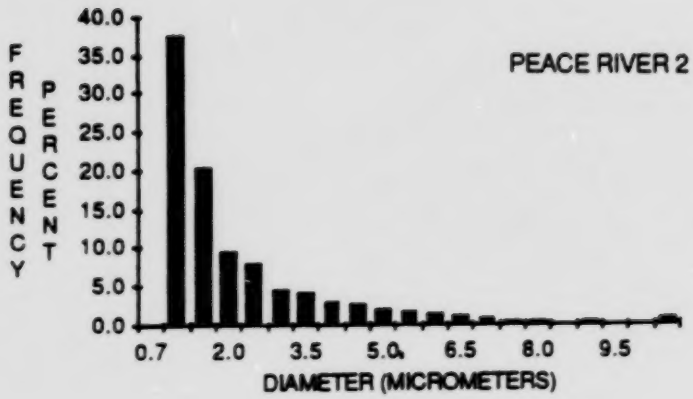
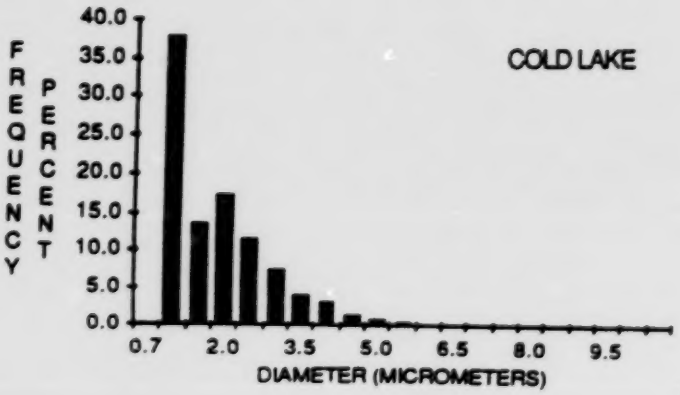


Figure 4.3 DROPLET SIZE DISTRIBUTION







4.2 Membrane Characterization and Selection

Successful separation of oil and water from an emulsion by a membrane is dependent on using a membrane which will preferentially permeate one component while excluding the other. In the case of a water in oil emulsion a membrane which would permeate oil and exclude water was desirable. This membrane would be oliophilic and hydrophobic. In general, this would be nonpolor material with no surface charge. The pore size should be smaller than the majority of the emulsified water droplets to physically exclude the water. For this study membranes which were commercially available in a variety of configurations were most desirable.

Table 4-2 summarizes the information collected and used for the membrane characterization and selection. Forty-two polymeric materials were initially identified as available as membranes or may be suitable for use as membranes. Although this list is long it is not exhaustive. Many polymers and mixtures of polymers have been investigated for use in porous membrane separations. In Table 4-2, the membrane materials are grouped as hydrophobic or hydrophilic as specified by the membrane manufacture. Also included in Table 4-2 is the surface charge. The surface charge was determined based on the known chemistry.

Where available films of these materials were obtained and the contact angle of water at the film surface measured. The contact angles are shown in Table 4-2. The contact angles measured ranged from 60° for polyvinyl fluoride to 116° for polyethylene/polyvinyl acetate. the lower contact angles indicate the material is hydrophilic while the higher contact angles indicate the material hydrophobic. Contact

TABLE 4-2 MEMBRANE SELECTION

MATERIAL COMPOSITION	MEMBRANE SUPPLIER	CONFIGURATION	FILM SUPPLIER	TRADE NAME	CONTACT ANGLE OF WATER (degrees)	SURFACE CHARGE
Hydrophilic material						
Cellulose Acetate	Desalination/DDS	Spiral/Flat/Tube/Fibre	Transilwrap	Transilwrap	69	Neutral
Cellulose Acetate Butyrate	NA		Eastman Kodak	UVEX	78	Neutral
Cellulose Tri-Acetate	Desalination/Dowex	Fiber/Spiral	Eastman Kodak	Kodacel	62	Neutral
Copolymer Acrylonitile/Vinyl pyrolidone	Daicel					Neutral
Ethylene-vinyl alcohol	Zenon	Flat Sheet				Neutral
Modacryl Polymer	Dorr-Oliver/Romicon	Fiber				Anionic
Polyacrylonitrile	Wafilin					Neutral
Polyamide	Kalle/Filmtec/Toray/Nitto	Spiral				Anionic
Polyaramid	Dupont	Fiber				Anionic
Polyetherimide			General Electric	Ultem	80	Neutral
Polyethersulfone S-hydrophilic	Zenon	Flat/Tube				
Polyimide	Berghof/Nuclepore	Flat	Dupont	Kapton		Neutral
Polyolefin-hydrophilic	Nitto					
Polyaromatic	Zenon	Tubular, Fiber				Anionic
Polysulphone -hydrophilic	Millipore/Zenon/DDS	Tube/Spiral/Fibre				Anionic
Polyvinylidene flouride-hydrophilic	Zenon	Tubular, Fiber				Neutral
Regenerated cellulose	Osmonics	Flat				Neutral
Vinyl formal	NA					
Nafion	Nafion		Du Pont	Nafion 117		Cationic
Hydrophobic material						
Polybenzimidazolone						None
Polyether ether Ketone	Zenon	Tube	ICI	Stabar K 200	86	None
Polyether Sulfone	Zenon,Patterson Candy	Tube	ICI	Stabar S 100	84	None
Polyethylene	NA	Tubular MF	Handiwrap	Handiwrap	94	None
Polyethylene-polyvinyl acetate	NA		Glad Cling Wrap	Glad Cling Wrap	116	None
Polypropylene	Celanese	Fibre MF				None
Polysulfone	Zenon, Millipore	Spiral, Tube, Fibre				None
Polytetrafluoroethylene	Millipore/ Aqua Chem	Spiral (Desal)	Du Pont	Teflon,PFA		None
Polytetrafluoroethylene	Millipore/ Aqua Chem		Du Pont	Teflon,FPE		None
Polyvinylidene Flouride	Abcor	Tubular	Pennwalt	Kynar	88	None
Not specified						
Co-Polyacrylonitrile/Methallyl Sulfonate	Rhone-Poulenc	Flat				
Polybenzzoxazindione, sulfonated	Bayer					
Polybutylene Terephthalate			General Electric	Valox		
Polycarbonate			General electric	Lexan	84	
Polyester			Dupont	Mylar	77	
Polyolefin	Nitto		Dow	Saranex	96	
Polypropylene / Polyolefin			Dupont	Clysar	94	
Polystyrene			Dow	Trycite	86	Neutral
Polyvinyl alcohol	Abcor	Tubular	Benton Plastic Co.	-		Neutral
Polyvinyl flouride			Dupont	Tedlar	60	Neutral
PVDC			-	-	75	Neutral
Saran FI - 120			-	-		
Stabilized Polyalcohol	Zenon			AVA	50°	Neutral

angles can only be determined for clean, non-porous surfaces. Unfortunately, it was impossible to obtain many of the potential membranes as non-porous films which limited this parameter's usefulness as a screening tool. Attempts were made to measure the surface contact angle of each of these films with a light mineral oil. However, due to the low surface energy of oil, it was impossible to make any accurate measurements using the tilting plate method.

Currently the number of applications for hydrophobic membranes is not as great as for hydrophilic membranes. Thus there is not as many commercially available hydrophobic membranes. Four hydrophobic polymeric membranes were selected for testing from the list in Table 4-2. They are polyether sulphone, polypropylene, polysulfone and polytetrafluoroethylene (Teflon). These membranes are all commercially available in a variety of pore sizes and configurations. Also included for testing is one hydrophilic polyaromatic membrane and ceramic membrane.

4.3 Oil Permeation Development Test

In the initial oil permeation tests, polyethersulphone, hydrophilic polyaromatic, polysulphone, polypropylene, Teflon and ceramic membranes were tested. Table 4-3 summarizes the results of these tests (detailed results are included in the appendix). The polyether sulphone and hydrophilic polyaromatic membranes did not produce any permeate. The 50K pore size of the polysulphone and the polypropylene produced a low flow of permeate while the smaller pore size for these membranes did not produce significant permeate. The Teflon and ceramic membranes which had larger pore sizes behaved better

Table 4.3

SUMMARY OF MEMBRANE SCREENING TEST RESULTS FOR OIL PERMEATION

Average Feed 2.2%

	Permeation Rate (GFD)	% H2O
Polyether Suphone 10K	no permeation	
Polyether Suphone 40K	no permeation	
Hydrophilic Polyaromatic 10K	no permeation	
Hydrophilic Polyaromatic 50K	no permeation	
Polysulphone 10K	very low	
Polysulphone 50K	low	1.1
Polypropylene 0.02μ (30K)	no permeation	
Polypropylene 0.04μ (50K)	low	1.6
Teflon 0.1μ (200K)	low	1.4*
Teflon 1.0μ	low	0.5*
Ceramic 1.0μ	0.27	1.2*

* average

with observably more permeate flow. The ceramic membrane permeate flow was measurable because the module used had significantly more surface area than the other cells used.

The permeates collected in these tests were analyzed for water content and the results are included in Table 4-3. In all cases the water content was lower than the feed. The lum Teflon membrane was the only membrane which produced a permeate with a water content low enough to meet pipeline requirements and thus treatment objectives. The fact that the polymeric membrane with the largest pore size produced the permeate with the lowest water content may be due to the fact that it had the highest flux rate. A similar phenomena has been observed in reverse osmosis whereby a solute permeates a membrane at a relatively slow rate while the solvent permeates at a faster rate which is controlled by the operating parameters. If the solvent permeation rate is decreased due to operating changes, the solute appears in the permeate at higher concentrations. In this observed case, the water may act as the solute and the oil like the solvent. Thus if the flux could be optimised the rejection would also be optimised. In subsequent testing, the oil permeation process was optimised using the Teflon membrane.

A series of six tests were carried out with the Teflon 1.0um membrane and the Teflon 0.5um membrane to determine optimum operating conditions. All optimization tests were carried out at temperatures between 40°C and 60°C. Higher temperatures would result in higher flux rates however, the manufacturer stated that at temperatures above 60°C the membrane will deteriorate and preliminary testing confirmed this fact. Details of the results of the optimization tests are included in

the Appendix B.

With the Teflon 1.0um membrane, the permeate quality was very good. The water content averaged 0.25%. The only time the amount of water in the permeate appeared to increase in the process was after the flow was adjusted. This could have resulted when the gel layer which forms on the membrane (a dynamic membrane) is cleared away with the increase in the flow. The optimum conditions which this system was run at with this membrane were 50°C and an inlet pressure of 90 psi. Under these conditions the permeate water content was 0.36% and the permeate rate 0.95 GFD.

The Teflon 0.5um membrane provided some very consistent results but the amount of moisture in the permeate was higher and the rate of permeation was approximately half of the 1.0um membrane.

Although the results show some promise in that the permeate quality is very good, the low flux rates will result in unfavourable economics for the process. The principal reason for the low flux rates is the very high viscosity of the emulsion. High viscosity reduces turbulence at the membrane surface resulting in a build up of water and sediment at the membrane surface preventing permeation.

The Reynolds number is an approximate calculation of the turbulence. It takes account the viscosity and the cell's shape and size. The Reynolds number for the 50°C emulsion in the flat sheet test cell used in these tests was 22. For turbulent flow the Reynolds number must be greater than 3000. Using traditional design (tube, flat sheet) turbulent flow cannot be obtained for these types of water-in-oil emulsions.

In addition to the effect of viscosity of the emulsion on the

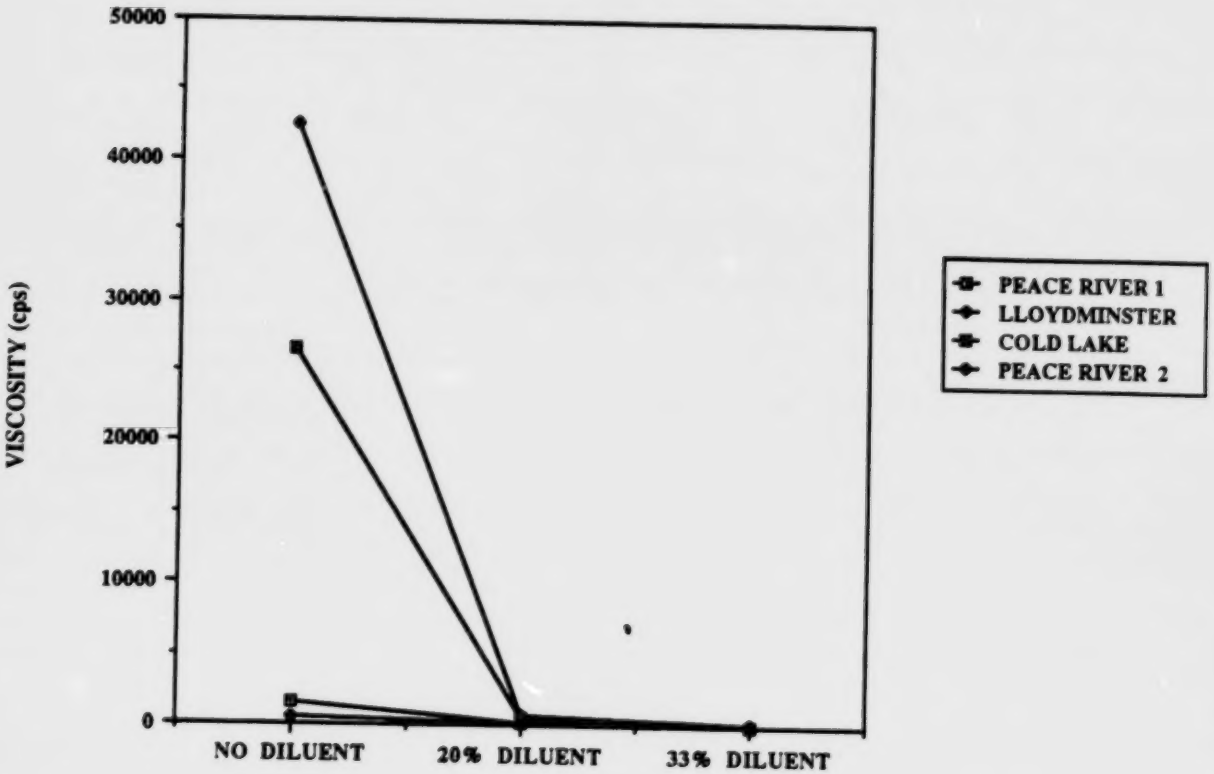
flow at the membrane surface the high viscosity of the oil will also limit the flux of the oil through the pores of the membrane.

In a standard half-inch tube UF-system the viscosity must be less than 7 cps for turbulent flow at standard velocities. A number of ways may be available to reduce the viscosity of the emulsion, the temperature of the feed could be increased. Figure 4-2 shows the viscosity temperature profile for the four emulsions examined in this study. At 80°C, the viscosity ranged from 55 cps for the Lloydminister emulsion (0.25% water) to 2100 cps for the Peace River 2 emulsion (37% water) which is too high for turbulent flow. Alternatively, a diluent could be added to the emulsion. Figure 4-4 shows the effect of dilution with mineral spirits on emulsion viscosity. Although the diluent dramatically decreased the viscosity, the viscosity with up to 33% diluent was still greater than 40 cps. Thus the flow would be laminar under standard operating conditions. Furthermore, considerable cost would be involved with recovery of the diluent after processing.

In summary, the conclusions drawn from the oil permeation tests are:

1. Ultrafiltration of water-in-crude oil emulsions can significantly reduce the water content of the oil.
2. A Teflon membrane with a 1.0um pore size reduced the water content of a 2.2% water in crude oil emulsion to less then 0.5% water which meets the treatment objectives with respect to quality.
3. The polysulphone, polypropylene and ceramic membrane tested reduced the water content by up to 50% but did not meet treatment objectives.
4. The hydrophilic polyaromatic and polyether sulphone membranes did

Figure 4.4 THE EFFECT OF DILUENTS ON BITUMEN



not permeate oil under the conditions tested.

5. The flux rates for the polysulphone, polypropylene, teflon and ceramic membranes were extremely low. Flux appears to be limited by the viscosity of the emulsion.

6. The optimum oil permeation conditions observed were as follows:

   Membrane: Teflon 1.0um

   Temperature: 53°C

   Applied pressure: 90 psi

   Velocity: 1.5 m/sec.

   Under these conditions the water concentration was reduced from 1.9% to 0.3% at a flux rate of 0.8 GFD.

4.4 Water Permeation Development Studies

The conditions necessary to form a stable oil-in-water emulsion from the Peace River 1 water in oil emulsion were determined. In the screening test, an oil in water emulsion was produced from a mixture of the Peace River 1 emulsion and water when the pH was greater than 9. At the point of emulsion reversal the colour changed from black to brown and the viscosity dropped dramatically. Temperature was not significant in the formation of a stable oil-in-water emulsion. The detailed results of this test are included in Table C-1 in the appendix.

A one to one ratio of water to Peace River 1 emulsion was titrated with sodium hydroxide and a stable oil-in-water emulsion was formed after addition of 7 millimoles OH^-/L of Peace River 1 emulsion. The end point of the titration was not absolute. As hydroxide was added droplets of oil suspended in the aqueous phase and as more

hydroxide was added the droplets became fewer and more difficult to settle. The complete set of observations for this test are included in Table C-2 in the appendix.

For permeation testing an emulsion was formed with one part Peace River 1 emulsion to three parts water and 7 millemoles/L sodium hydroxide. This emulsion was concentrated in an ultrafiltration trial, where the permeating water was removed. Figure 4-5 shows the flux rate as a function of oil concentration. The flux rate of each of the membranes, tested dropped very rapidly as the oil concentration increased from 30% to 60%. The HST and HSC membranes had slightly higher flux rates than the HEM membrane.

Upon completion of the concentration trial additional dialysis water was added to the concentrate to dilute the feed and a stability trial run for 4 hours. Figure 4-6 shows the permeate rate over the duration of the stability trial. As shown in this figure the permeate rates for each of the membranes tested were stable over this period.

Metals were analysed in the water added for dialysis and in the permeate from the concentration trial to determine if metal salts were being washed out of the oil by this process. Table 4-4 shows the results of this analysis. Phosphorus, sulphur and vanadium were washed out of the oil. However, the concentrations of the majority of the other metals were decreased in the permeate indicating they were left in the concentrated oil stream. Although some metals were extracted, this process is not efficient at the pH necessary to maintain a stable emulsion for testing.

A specific method for measuring surfactants in the permeate

FIGURE 4-5 WATER PERMEATION TEST: CONCENTRATION TRIAL

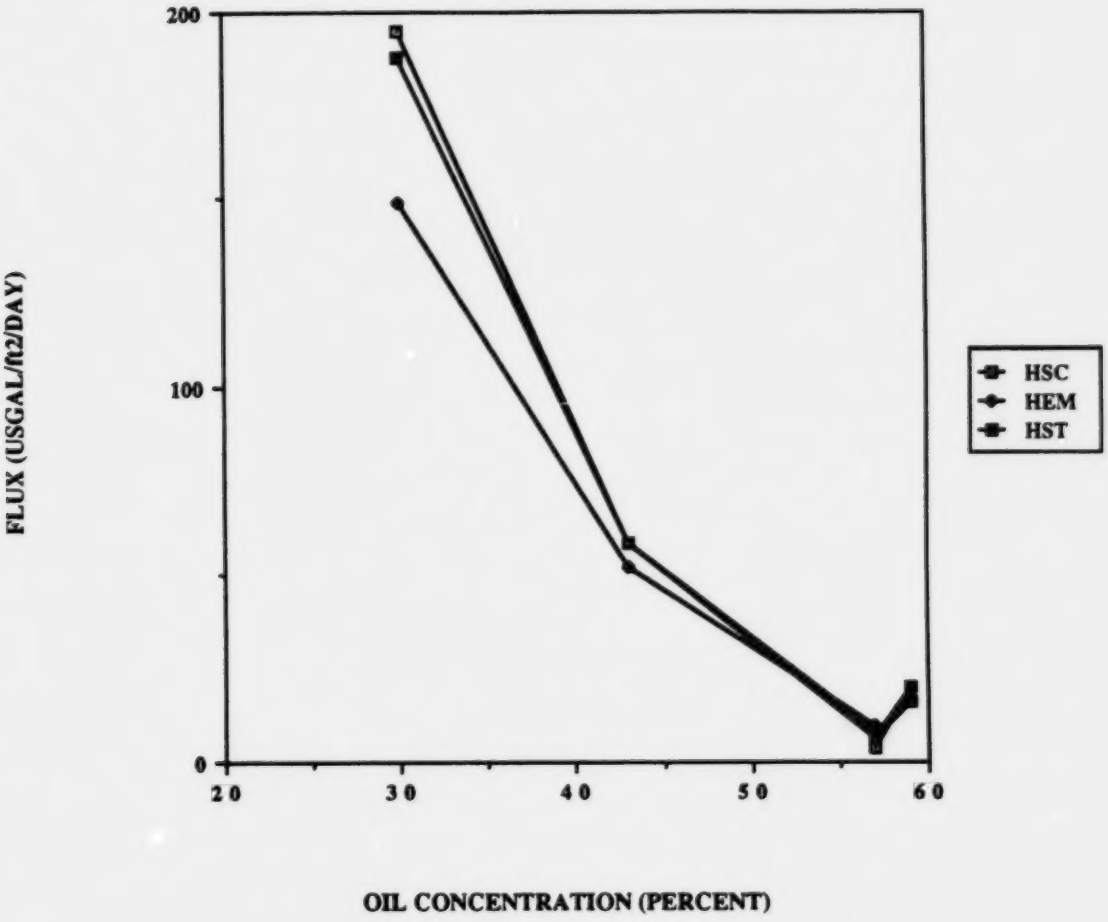


FIGURE 4-6 WATER PERMEATION TEST: STABILITY TEST

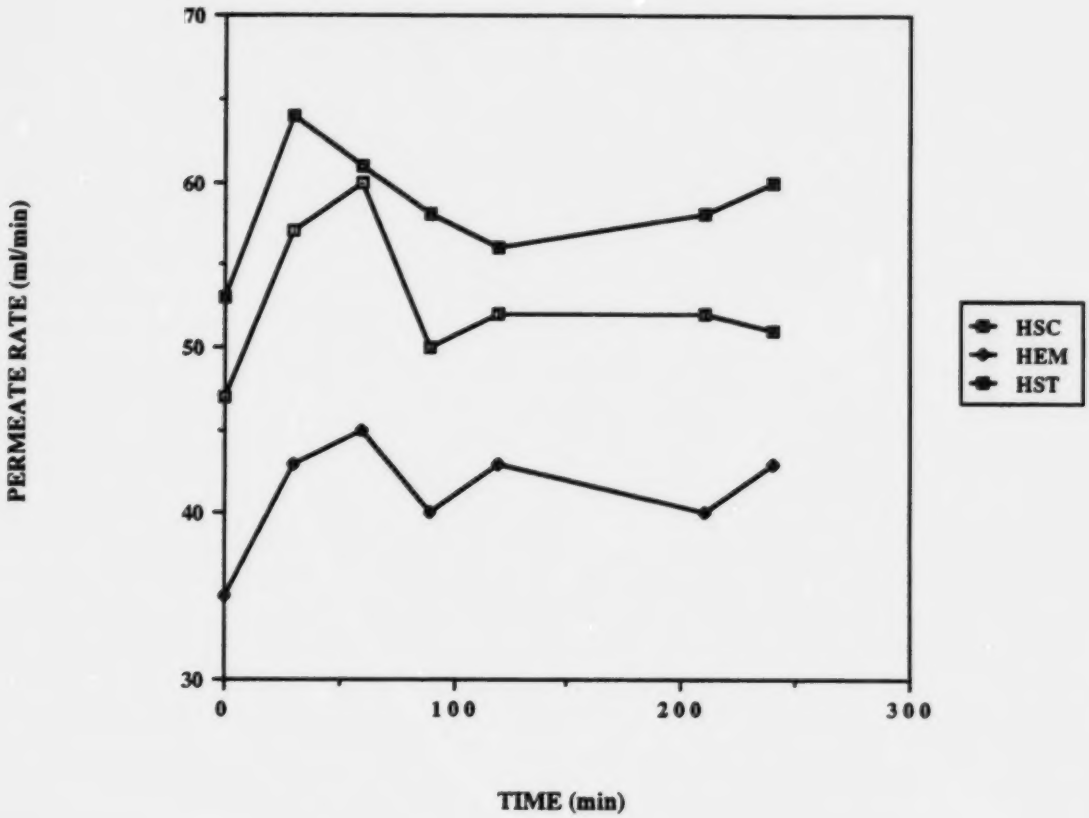


TABLE 4.4 EXTRACTION OF METALS BY DIALYSIS

(mg/kg)	DIALYSIS WATER	AVERAGE PERMEATE	INCREASE OR (DECREASE)
Calcium	35	0.94	(34.06)
Magnesium	8.1	0.30	(7.80)
Sodium	240	155.00	(85.00)
Potassium	1.9	1.40	(0.50)
Aluminum	0.71	0.27	(0.44)
Barium	0.024	0.01	(0.02)
Beryllium	< 0.001	<0.001	-
Boron	< 0.1	<0.15	-
Cadmium	< 0.002	< 0.002	-
Chromium	0.16	0.32	0.16
Cobalt	< 0.01	< 0.01	-
Copper	0.086	0.02	(0.06)
Iron	1.5	0.23	(1.27)
Lead	< 0.04	< 0.04	-
Manganese	0.023	<0.006	(0.02)
Molybdenum	< 0.02	0.04	0.02
Nickel	0.069	0.02	(0.04)
Phosphorus	< 0.1	2.95	1.95
Silicon	1	0.72	(0.28)
Silver	< 0.01	< 0.01	-
Strontium	0.17	0.01	(0.16)
Sulphur	9.8	50.00	40.20
Thallium	< 0.2	< 0.2	-
Titanium	0.055	0.04	(0.02)
Vanadium	< 0.01	0.03	0.00
Zinc	0.073	0.04	(0.04)
Zirconium	< 0.01	< 0.01	-

was not available thus the extraction of surfactant could not be directly measured. However, observation of the concentrated oil-in-water emulsion produced by processing indicated that the emulsion was stable and the surfactants and salts stabilizing the emulsion had not been effectively extracted.

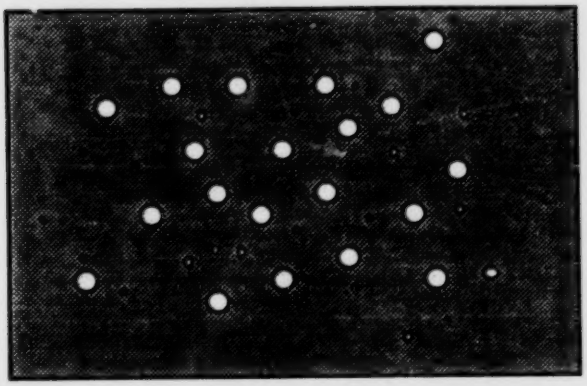
The poor extraction of surfactant and metals suggested that the water held in the original emulsion was not released. This indicated that the emulsion had not "reversed" but the "oil" droplets in the newly formed oil in water emulsion were droplets of water-in-oil emulsion. A sample of the "oil-in-water" emulsion was examined microscopically and visually this appeared to be the case. Figure 4-7 shows the observed "oil in water" emulsion schematically. F. Mouritz of Saskatchewan Research has been studying the formation of oil-in-water emulsions from water in oil emulsions for the purpose of facilitating pumping and has observed similar results (Mouritz, 1989).

In summary, the conclusions drawn from the water permeation tests are:

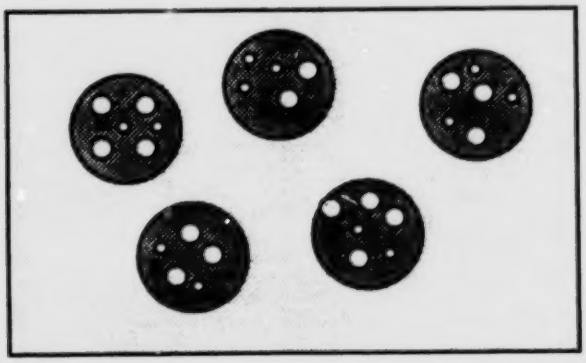
1. HSC, HST and HEM ultrafiltration membranes effectly permeate water from a stable oil-in-water emulsion and produce a permeation rate which is stable with time.

2. Addition of water and sodium hydroxide to a water in crude oil emulsion with controlled mixing can produce a stable emulsion of that emulsion in water.

FIGURE 4.7 OIL IN WATER EMULSION FORMATION



WATER IN OIL EMULSION



OIL IN WATER EMULSION
FORMED FROM WATER
IN OIL EMULSION

WATER

BITUMEN

3. Addition of water and sodium hydroxide to a water in crude oil emulsion to produce a stable emulsion and subsequent removal of the water by ultrafiltration is not an effective means of extracting the surfactants and salts and producing a clean water-free oil.

4.5 Oil Field Emulsion Tests

Oil permeation trials were run for each of the oil field emulsions obtained. The physical/chemical characteristics of the "as received" emulsions are presented and discussed in Section 4.1. Table 4-5 summarizes the results of the oil permeation trials. Detailed results are included in Table D-7 to D-9 in the appendix.

In each of the three trials the permeate produced had water concentration lower than 0.5% and thus meets treatment objectives. Unfortunately, the flux rates were extremely low. These results confirm the conclusions made based on the oil permeation development tests using the Peace River 1 emulsion.

The conditions necessary to form a stable oil-in-water emulsion from each of the water-in-oil emulsions. Sodium hydroxide was added incrementally to a mixture of 100 mL of Lloydminster emulsion and 100 mL of water with controlled mixing. The pH was increased to 14 without an emulsion forming. In another mixture of Lloydminster emulsion and water, sulphuric acid was added incrementally with mixing to pH 1 and an emulsion was not formed. The same procedures were followed with a sample of the Cold Lake emulsion. An oil-in-water emulsion could not be formed with this sample either. On the other hand, when procedures were followed with the Peace River 2 emulsion a

TABLE 4-5 OIL PERMEATION TEST RESULTS FOR OIL FIELD EMULSIONS

SOURCE	AVERAGE PERMEATE RATE (USGAL/ft2/DAY)	PERCENT MOISTURE	
		FEED	PERMEATE
LLOYDMINSTER	6.08	4	0.18
COLD LAKE	0.24	19	<0.1
PEACE RIVER 2	<0.001	16	0.5

stable emulsion was formed upon addition of 14.5 millimoles of OH^-/L of emulsion. The reason that a stable emulsion was formed upon addition of hydroxide to the Peace River emulsion samples but not the Cold Lake or Lloydminster samples can not be defined by the data obtained during this study. However, it can be inferred that there exists water soluble surfactants in the Peace River emulsion that are activated by increasing pH. These surfactants do not exist in the Cold Lake and Lloydminster emulsions. The type of surfactants in a crude oil emulsion are site specific and dependent on the nature of the crude oil and the process used to extract the oil. Stable oil-in-water emulsions of the Cold Lake and Lloydminster emulsions may be formed by addition of other emulsifying agents.

The Peace River 2 oil-in-water emulsion was processed under the same conditions used in the development studies. Figure 4-8 shows the flux rate as a function of concentration. This figure shows that for all three membranes tested the flux rate dropped rapidly between 30 and 40% oil. The concentrate resulting from this trial was a stable 40% oil-in-water emulsion. The results of the stability test are shown in Figure 4-9. This figure shows that the permeate rate was stable for each membrane tested. The HSC membrane had a higher flux rate than the other two membranes.

In summary the conclusions drawn from the oil field emulsions tests are:

1. Ultrafiltration of water-in-oil crude oil emulsions with a Teflon 1.0um membrane can reduce water concentrations from 20% water to less than 0.5% water, however the flux rates are very low (less than 6 gfd).

FIGURE 4-8 PEACE RIVER 2 WATER PERMEATION CONCENTRATION TRIAL

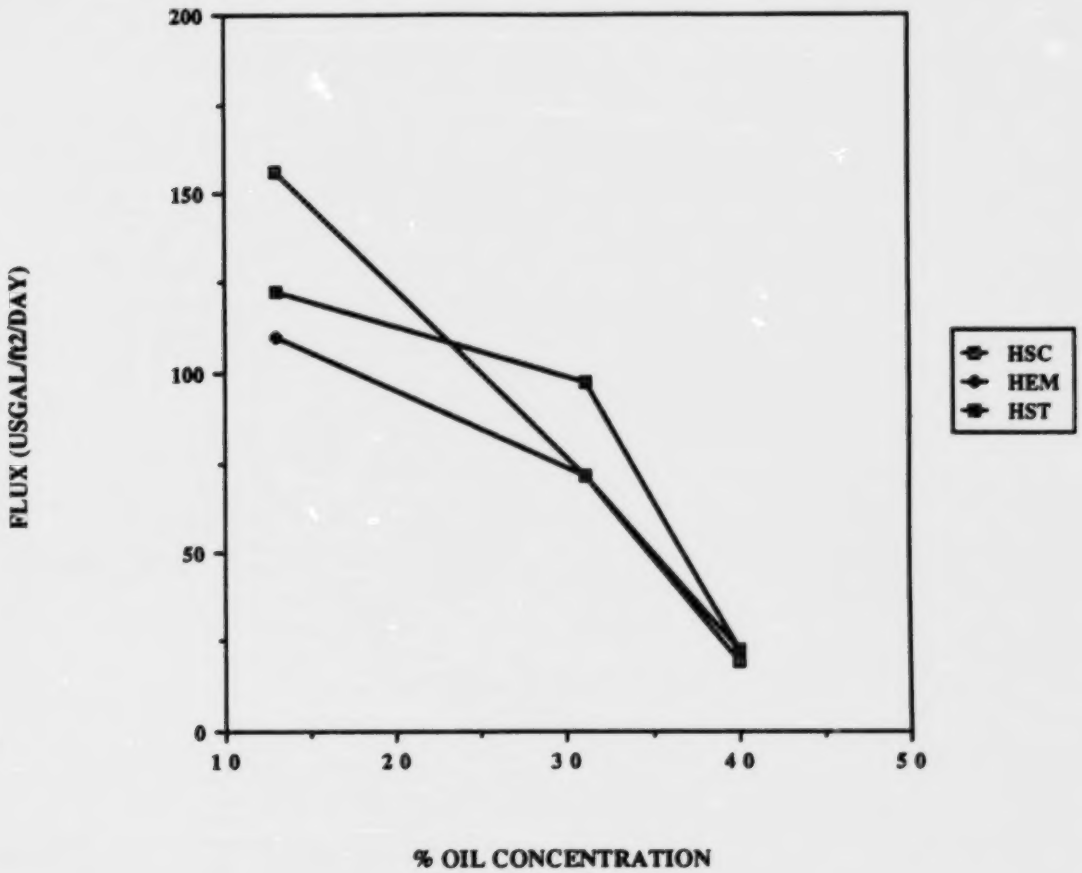
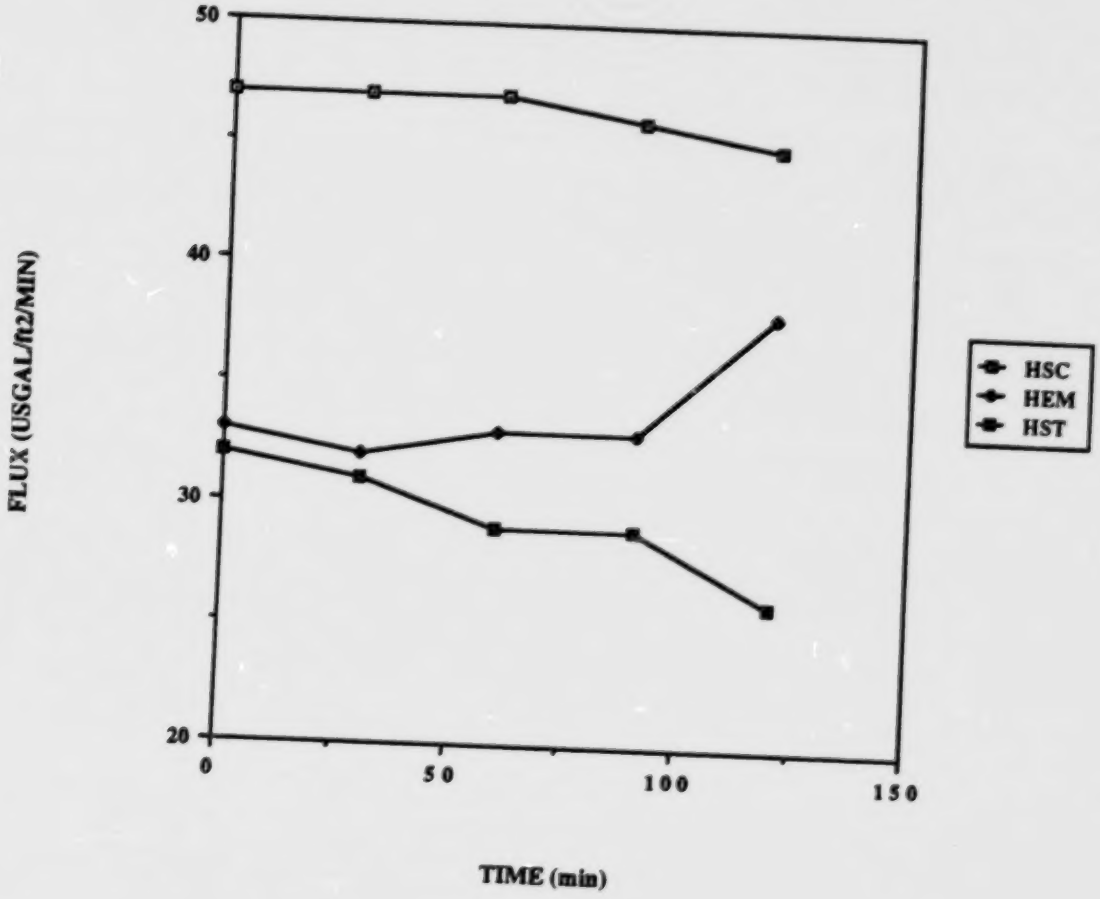


FIG 4-9 PEACE RIVER 2 WATER PERMEATION STABILITY TRIAL



2. Addition of water and sodium hydroxide to a water in crude oil emulsion can produce a stable emulsion however the effect is site specific.

3. Addition of water and sodium hydroxide to a water in crude oil emulsion to produce a stable emulsion and subsequent removal of the water by ultrafiltration is not an effective means of extracting the surfactants and salts and producing a clean water-free oil.

REFERENCES

Barnett, Jack, W. (1988) "Desalting can Remove More Than Salts and Sediments", Oil and Gas Journal. April 11, 1988, pg. 43 - 49.

Mouritz, Frank, (1989). Saskatchewan Research, Personal Communication

APPENDIX A

EXPERIMENTAL PROCEDURES

A-1 Particle Size Analysis of Water Droplets in Four Bitumen Samples

The 3 oil field Bitumen samples Lloydminster (83-02), Cold Lake (80-05) and Peace River (02-01) were analysed for their water droplet size and distribution. Along with these samples, the original Peace River sample was analysed (01-01).

1. 01-01 Peace River 1
2. 02-01 Peace River 2
3. 80-05 Cold Lake
4. 83-02 Lloydminster

METHOD:

A small portion of the samples 01-01 and 83-02 were placed on a microscope slide and a coverslip was set on top. The samples were viewed in transmitted illumination on the optical microscope at a magnification of 1250X. Images of the droplets were transferred via the video camera to the television monitor of the LeMont OASYS Optical Analysis System. Equivalent circular diameters of the droplets were automatically calculated by the image analyser. Samples 02-01 and 80-05 could not be spread out as thinly on the microscope slide, and therefore had to be viewed at a magnification of 315X to allow for sufficient illumination.

The microscope slides were viewed in transmitted illumination on the optical microscope and analysed on the LeMont OASYS Optical Analysis System.

A-2 Viscosity Tests

The viscometer used during these tests was a Brookfield LVT. For each sample, 400 mL of bitumen was placed in a 600 mL beaker. Being viscosity is dependent on temperature, all the samples were kept in a water bath and allowed ample time to equilibrate with the water baths temperature.

The Brookfield LVT has several different spindles for testing viscosity. In our tests spindle 4 was used for high viscosity readings and spindle 2 was used for low viscosity reading. At all times the spindle guard was in place.

For instruction on how to use the viscometer refer to chapter 3 of the Brookfield LVT handbook.

A-3 Contact Angle Measurement

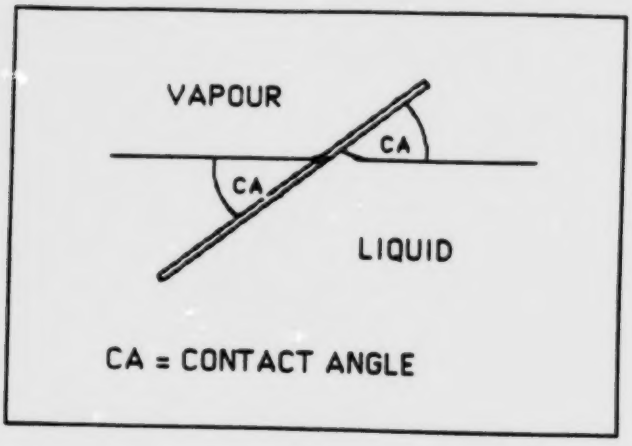
The tilt plate method was used to measure the contact angles. The materials tested were in the form of a film, being the pores in the membranes would give false results.

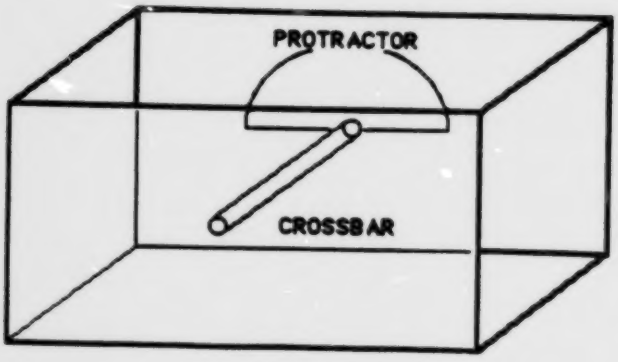
The equipment used consisted of a pivoting plate with a protractor attached, to measure the angles. This set up was secured in a container of water, where the water level was just below the pivoting point (or the base of protractor).

To measure the contact angle the film was secured to the plate and rotated to the point where the water met the film with a flat meniscus.

(Refer to Fig. A-1)

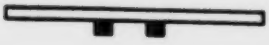
FIGURE A-1 Appearance of the miniscus at various angle settings



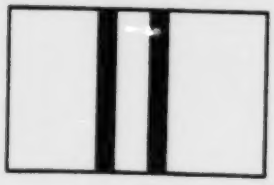


FILLED UP TO THE BASE LINE OF THE PROTRACTOR

SIDE VIEW OF GLASS PLATE



UNDERSIDE VIEW OF GLASS PLATE



MEMBRANE FILM IS CAST ONTO THE TOP SIDE OF THE GLASS PLATE. GLASS OR PLASTIC BARS (BLACK) ARE ATTACHED TO THE BOTTOM SUCH THAT THEY ARE SLIGHTLY FURTHER APART THAN THE WIDTH OF THE CROSS BAR (OPPOSITE) TO ALLOW FREE MOTION. THE PLATE IS ROTATED UNTIL THE LIQUID LEVEL AT THE MEMBRANE/ LIQUID INTERFACE APPEARS PERFECTLY FLAT. THE ANGLE IS THEN READ OFF THE PROTRACTOR

A-4 Description of the Six Tube Module Tests

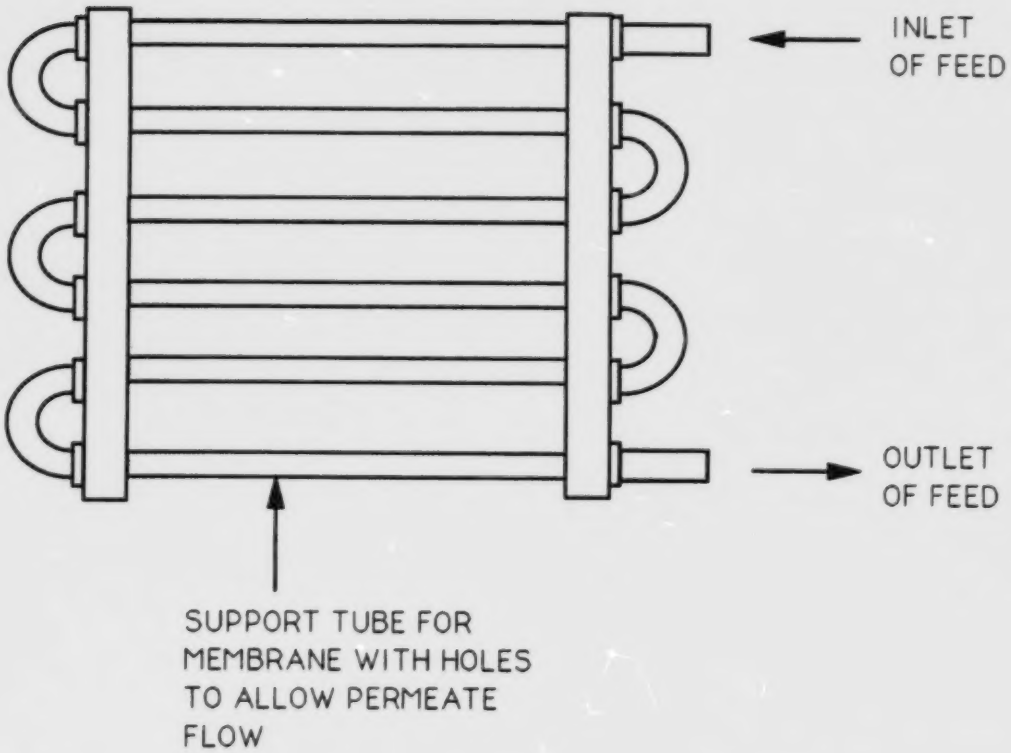
This module is made of stainless steel and acts as support for one foot 1/2" membrane tubes. Of the one foot membranes only 10 3/4" of the tubes are exposed to the feed, the reason for this is there are 3/4" seals at the end of each tube to keep the module from leaking.

Each of the stainless steel support tubes has holes in them to allow the permeate to flow through. The permeates are caught in separate troughs and each one has a sampling tube. Being the tubes are in series, the pressure on the inlet side is more then on the outlet side; therefore this has to be considered when reviewing the results.

The membranes were usually run at a temperature of 40°C and at pressures less then 90 psi.

(Refer to Fig. A-2)

FIG A-2 TUBULAR SIX MEMBRANE MODULE TESTER



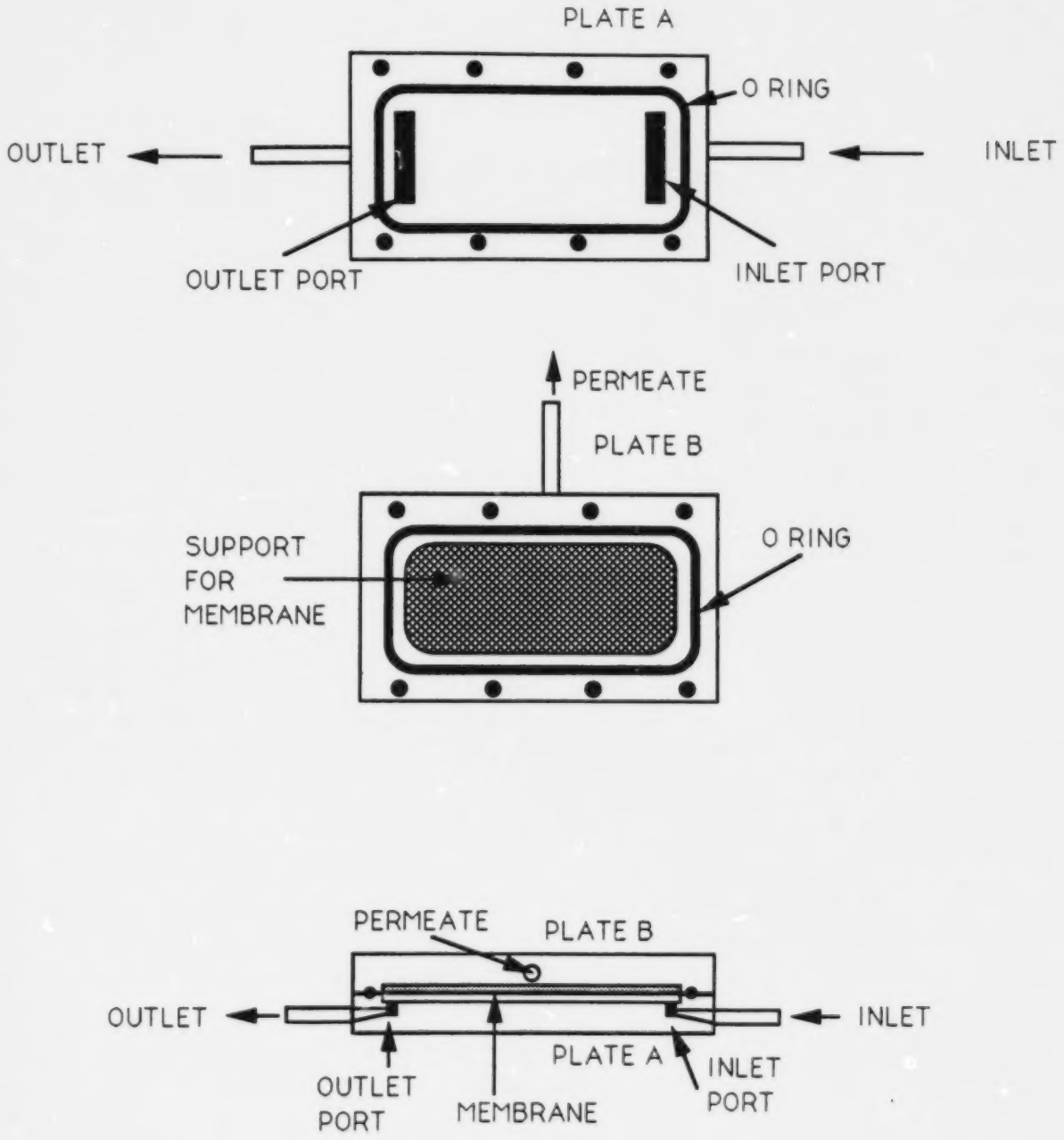
A-5 Description of the Flat Sheet Module Tester

The cell is made up of two 1" stainless steel plates 4 1/2" X 7 3/4". One plate has inlet and outlet lines and inlet and outlet ports machined into it. The second plate has a porous stainless steel insert which supports the membrane and allows the permeate to flow through it to the permeate tube. These plates are sealed with an "O" ring and 8 bolts.

The membrane surface area is 2" X 5" (10 inch^2) and it was run at pressures less then 100 psi for ultra-filtration applications.

(Refer to Fig. A-3)

FIG A-3 FLAT SHEET MODULE TESTER



A-6 Analytical O/W Methodologies

I. Karl Fisher Moistures

(i) Initial Method

100 mg of sample was dissolved in 2 mLs of chloroform. The solution was then transferred to a Karl Fisher titration cell containing 5 mLs of methanol and titrated potentiometrically.

(ii) Revised Method

1 g of sample was dissolved in 10 mLs of 25% isopropanol in toluene. 100 uLs of solution was then introduced into a Karl Fisher titration containing 5 mLs of methanol and titrated potentiometrically.

II. Oil & Grease

Depending on the sample, 1 mL to 100 mLs of sample were measured and made to 500 mLs with reverse osmosis deionized water (RODI). The sample was acidified to pH 2 with HCL and then extracted with 50 mLs of dichloromethane. The extract was passed through an Allihn filter containing Na_2SO_4 into a 500 mL flat bottom flask. Two more 50 mL aliquots of dichloromethane were processed as above. The combined extract was rotovapped to approximately 5 mLs and then transferred to a preweighed 10 mL vial and taken to dryness with a gentle stream of nitrogen.

III. Metals

1 g of sample was digested with 5 mLs of concentrated nitric acid in a teflon pressure vessel in a microwave for 10 minutes. The solution was then transferred to a graduated centrifuge tube and made to 50 mLs with RODI water the solution was then analysed using inductively coupled plasma (ICP).

IV. pH

A glass electrode was calibrated against standard buffers (pH 6.00 and pH 10.00). The samples were then measured after manually stirring for 5 minutes. The probe was cleaned with toluene and then reequilibrated with tap water between measurements.

V. Conductivity

The conductivity probe was calibrated against standard KCL solutions and then samples were measured. The probe was cleaned with toluene and reequilabrated with tap water between measurements.

VI. Sediments

A weighed sample (approximately 10 grams) was dissolved in toluene and then passed through a preweighed GFC filter with subsequent toluene rinses. The filter was then dried at 60°C to constant weight.

APPENDIX B

RAW RESULTS OIL PERMEATION STUDY

TABLE B-1 VISCOSITY OF THE PEACE RIVER 1 WATER IN OIL (W/O) EMULSION AT VARIOUS TEMPERATURES

SAMPLE	TEMP. (deg. cel.)	SPINDLE MODEL AND NUMBER	SPEED	FACTOR	RANGE	READING	VISCOSITY (cps)	AVERAGE
Standard (55040cps @ 25 deg. cel.)	24	LV 4	6	1000	100 000	a) 61.1 b) 59.6	61100 59600	60400
W/O EMULSION	24	LV 4	60	100	10 000	a) 33.8 b) 34.6	3380 3460	3420
W/O EMULSION	40	LV 4	60	100	10 000	a) 9.2 b) 9.8	920 980	950
W/O EMULSION	22	LV 2*	6	50	5000	a) 73.4 b) 73.5	3670 3675	3673
W/O EMULSION	40	LV 2	30	10	1000	a) 55.8** b) 62.0	558 620	589
W/O EMULSION	60	LV 2	60	5	500	a) 35.8 b) 42.8	179 214	197
W/O EMULSION	80	LV 2	60	5	500	a) 14.8 b) 17.0	74 85	80
W/O EMULSION	100	LV 2	60	5	500	a) 8.4 b) 8.3	42 41.5	42

* Due to a low scale reading (< 10) of the previous measurement a larger spindle was chosen to increase acurracy

** Temperature fluctuation increased with an increase in temperature affecting reproducibility of certain results

NOTE : The container used to measure the viscosity of the sample was a 600 ml beaker.
In all cases the spindle guard was used for the viscosity measurements.

TABLE B-2 FLUX TESTS FOR OIL PERMEATION ON TUBULAR MEMBRANES

august 19 1988 Fluxrates (mL/min.)

Conditions
Feed mineral spirits
Temperature 23 C
Flowrate 20 L/min.
Configuration 1/2" straw

Pressure Inlet	Pressure Outlet	*Position* / *Membrane*	1 PESMA polyethersulphone 10K	2 ALT 13V17 Hydrophilic Polyaromatic 10k	3 PSC11 polysulphone 10k	4 T102 polysulphone 50k	5 ALT 1507 Hydrophilic Polyaromatic 50k	6 AET polyethersulphone 40K
50	30		NM	NM	1	6	1	1

august 24 1988 Fluxrates (mL/min.)

Conditions
Feed PEACE RIVER 1
Temperature 60 C
Flowrate 12L/min
Configuration 1/2" straw

Pressure Inlet	Pressure Outlet	*Position* / *Membrane*	1 PESMA polyethersulphone 10K	2 ALT 13V17 Hydrophilic Polyaromatic 10k	3 PSC11 polysulphone 10k	4 T102 polysulphone 50k	5 ALT 1507 Hydrophilic Polyaromatic 50k	6 AET polyethersulphone 40K
50	15		no perm.	no perm.	NM	NM	no perm.	no perm.
100	25-30		no perm.	no perm.	NM	NM	NM	NM

august 25 1988 Fluxrates (mL/min.)

Conditions
Feed PEACE RIVER 1
Temperature 76 C
Flowrate 13 L/min.
Configuration 1/2" straw

Pressure Inlet	Pressure Outlet	*Position* / *Membrane*	1 PESMA polyethersulphone 10K	2 ALT 13V17 Hydrophilic Polyaromatic 10k	3 PSC11 polysulphone 10k	4 T102 polysulphone 50k	5 ALT 1507 Hydrophilic Polyaromatic 50k	6 AET polyethersulphone 40K
100	35		no perm.	no perm.	no perm.	NM	no perm.	no perm

*reversed inlet and outlet such that position 6 "sees" 100psi

100	35	no perm.	no perm.	no perm.	NM (SAMPLED)	no perm.	no perm

NM-not measurable

TABLE B-3 FLUX TESTS FOR OIL PERMEATION ON FLAT SHEET MEMBRANES

September 8 1988 **Flux (mL/min.)**

Conditions
Feed PEACE RIVER 1
Temperature 55 - 60 °C
Flowrate 2 L/min.
Configuration NRC TEST CELLS

Pressure Inlet	Pressure Outlet	*Position* *Membrane*	1 Celguard 2400 Polypropylene .02u (30k)	2 K750 Teflon 1.0u	3 Paper Blank	4 K150 Teflon 0.1u (200k)	5 Celguard 2500 Polypropylene .04u (50k)
30	6		no perm.	fluxed-NM	fluxed/fouled	no perm.	no perm.
50	20		no perm.	fluxed (4x K150)	fluxed/fouled	fluxed NM	no perm.
75	45		no perm.	fluxed NM	fluxed/fouled	fluxed NM	no perm.
100	64		no perm.	fluxed-NM	fluxed/fouled	fluxed NM	fluxed- NM

September 11 1988 **Flux (mL/min.)**

Conditions
Feed PEACE RIVER 1
Temperature 75-80°C
Flowrate 2 L/min.
Configuration NRC TEST CELLS

Pressure Inlet	Pressure Outlet	*Position* *Membrane*	1 Polypropylene .02u (30k)	2 Teflon 1.0u	3 Paper Blank	4 Teflon 0.1u (200k)	5 Polypropylene .04u (50k)
30	9		no perm.	fluxed-NM	fluxed/fouled	fluxed-NM	no perm.
50	23		no perm.	fluxed-NM	fluxed/fouled	fluxed-NM	no perm.
75	48		no perm.	fluxed-NM	fluxed/fouled	fluxed-NM	fluxed-NM
100	66		no perm.	fluxed-NM SAMPLED	fluxed/fouled	fluxed-NM SAMPLED	fluxed-NM SAMPLED

TABLE B-4 ANALYTICAL RESULTS FROM MEMBRANE TESTING

DATE TESTED	% WATER	SAMPLE DISCRIPTION
AUG 25/88	1.9	FEED (ORIGINAL)
AUG 25/88	1.1	PERMEATE FROM THE POLYSULPHONE MEMBRANE IN POSITION 4 (1.6ML/HR)
SEPT 8/88	1.3	PERMEATE FROM THE K150 TEFLON MEMBRANE IN POSITION 4
SEPT 8/88	0.33	PERMEATE FROM THE K750 TEFLON MEMBRANE IN POSITION 2
SEPT 11/88	2.4	FEED (ORIGINAL)
SEPT 11/88	1.4	PERMEATE FROM THE K150 TEFLON MEMBRANE IN POSITION 4
SEPT 11/88	0.63	PERMEATE FROM THE K750 TEFLON MEMBRANE IN POSITION 2
SEPT 11/88	1.6	PERMEATE FROM THE CELGUARD 2500 MEMBRANE IN POSITION 5 (POLYPROPYLENE)

TABLE B-5 OIL PERMEATION OPTIMIZATION WITH TEFLON 0.5μ MEMBRANE

Date	Temp. (°C)	Time (min)	Inlet. Press. (psi)	Outlet Press. (psi)	Feed Flowrate (L/min)	Permeate Rate (mL/min)	Permeate Rate (GFD)	Feed Conc. (% Water)	Permeate Conc. (% Water)	Comments
Oct. 20	40	60	40	17	7.5	0.33	1.89	3.3	2.7	
Oct. 20	40	120	50	20	7.5	0.33	1.87		1.6	-Mb failed (high pressure) (replaced pressure gauges)
Oct. 31	40	60	40	10	7	0.17	0.94	2.9	1.2	
Oct. 31	40	120	50	20	7	0.15	0.85		0.86	
Oct. 31	40	180	50	10	9	0.15	0.85		1.4	
Oct. 31	40	240	60	25	7	0.13	0.75		0.49	
Nov. 1	40	60	60	10	9	0.08	0.47	2.4	<0.1	
Nov. 1	40	90	40	10	7	0.03	0.19		0.52	
Nov. 1	40	120	40	10	7	0.08	0.47		0.46	-Removed the membrane
Nov. 1	40	150	70	40	7	0.13	0.75		0.21	and cleaned in mineral spirits
Nov. 1	40	180	70	13	9	0.08	0.47		0.32	
Nov. 1	42	210	80	50	7	0.12	0.66		0.55	

TABLE B-6 OIL PERMEATION OPTIMIZATION WITH TEFLON 1.0μ

Date	Temp (°C)	Time (min)	Inlet Press. (psi)	Outlet Press. (psi)	Feed Flowrate (L/min)	Permeate Rate (mL/min)	Permeate Rate (GFD)	Feed Conc. (% Water)	Permeate Conc. (% Water)	Comments
Oct 7	40	60	40	10	7	0.58	3.30	3.6	1.4	
Oct 7	41	120	60	12	9	0.58	3.30		0.65	
Oct 7	41	130	60						0.77	Mb failed (high pressure)
Oct 12	42	60	40	15	7.5	0.42	2.36	2.1/1.5	0.76	
Oct 12	46	120	60	25	7.5	1.33	7.54		1.2	
Oct 12	46	180	60	30	22	40.00	226.20		9.5	Mb failed (high pressure) (pressure gauges replaced)
Nov 2	40	60	40	20	7	0.30	1.70	1.9	<0.1	
Nov 2	40	120	50	30	7	0.13	0.75		<0.1	
Nov 2	40	0.18	50	12	9	0.08	0.47		0.59	
Nov 2	40	240	60	38	7	0.08	0.47		0.43	
Nov 2	40	300	60	12	9	0.08	0.47		<0.1	
Nov 2	40	330	70	40	7	0.08	0.47		<0.1	
Nov 2	53	345	95	80	7	0.17	0.94		0.41	
Nov 2	53	365	90	70	7	0.17	0.94		0.31	

TABLE B-7 OIL PERMEATION OPTIMIZATION WITH A CERAMIC MEMBRANE

Date	Temp. (°C)	Time (min)	Inlet. Press. (psi)	Outlet Press. (psi)	Feed Flowrate (L/min)	Permeate Rate (mL/min)	Permeate Rate (GFD)	Feed Conc. (% Water)	Permeate Conc. (% Water)	Comments
Nov. 3										Mb produced no permeate
Nov. 4	53	60	80	50	5	0.07	0.11	1.9	1.6	
Nov. 4	63	180	80	20	5	0.05	0.08		-	
Nov. 4	68	240	75	50	5	0.08	0.13		0.58	
Nov. 4	70	300	85	60	6	0.10	0.16		1.5	
Nov. 4	72	360	90	70	6	0.12	0.19		0.91	
Nov. 7	79	450	90	70	4	0.12	0.19		1.3	79° C is Max. with Heater
Nov. 7	79	495	90	70	4	0.15	0.24		1.4	
Nov. 7	79	530	90	14	10	0.17	0.27		-	

APPENDIX C

RAW RESULTS WATER PERMEATION STUDY

TABLE C-1 EFFECTS OF pH AND TEMPERATURE ON DILUTED BITUMEN

Sample	Temperature ° C the mix. was heated to	pH of Water added	Comments 1 to 1 oil and water
1	20	7	- After the solution was well mixed layers still formed, it appeared that no water was taken up into the oil layer.
2	20	8	- Results were the same as sample 1 but the bottom was a bit brown in colour.
3	20	9	- There was 2 layers, oil on top and water on the bottom. When it was initially mixed it appeared homogeneous, but layering occcured in 10 minutes. The top layer was still black but the bottom layer was a dirty creamy brown. The bottom layer was only 12-13 ml, therefore 8 ml were consumed into the bitumen.
4	20	11	- The results were the same as sample 3, however when the solution was mixed it was brown, with a low viscosity and it took several hours to separate.
5	80	7	- after mixing and heating the solution did not layer after 24 hours. In a watch glass it appeared homogeneous, but it was very viscous. NOTE there were several mls of water in the bottom of the beaker.
6	80	8	- The results were the same as sample 5
7	80	9	- The bottom layer was slightly coloured but it was only 10 ml, the top layer was very viscous.
8	80	11	- After 24 hours the bottom layer was 10 ml and it was the colour of sample 3 and 4. The top layer also had the same properties as these samples.

TABLE C-2 TITRATION OF BITUMEN AND WATER

- To determine the amount of NaOH needed to break the water in oil emulsion a titration was performed on 100 ml of tap water and 100 ml of bitumen with 0.125 N NaOH, while stirring with a magnetic stirrer.

Volume of 0.125 N NaOH (ml)	Comments
0	- After the solution was mixed well the mixture still seperated (black emulsion on top)
1	- The oil started to break up, the droplets were very small and suspended, but there was still 2 layers.
1.25	- Mixture still separated and the droplets may be smaller.
1.9	- Same as above
2.5	- Same as above
2.9	- Same as above
3.4	- The mixture still separated, however it took 5 to 10 minutes. The top layer was still very black.
3.8	- Same as above
5.1	- Solution is black right through and it has a low viscosity.
5.5	- After the NaOH was added, the mixture sat for 35 minutes, it did not seem to seperate and it was black right through. There appeared to be droplets of oil or water mixed through the solution.
6 to 13 (at 0.5 ml intervals)	- The solution is still very thin, the top of the vortex has a shine or sheen like oil on water.
13.6	- The sheen is more apparent and the solution is brown right through after mixing, but when the solution sat for 1 hour it separated into 2 layers black on top and a 70 ml brown bottom layer. - It was at this point that I thought the solution was oil in water even though it separated it would mix very easy, and in the process of removing the water it would remain homogeneous.

NOTE: It is difficult to determine the exact reversal point; we felt it occured when the viscosity of the solution was similar to water and it turned from black to brown. At this point a sheen appeared on the surface of the vortex.

TABLE C-3 CONTINUED BENCH TESTS ON THE TITRATED SAMPLE

-Following the titration, the solution with 2 layers was mixed to make it homogeneous and then it was separated into 2 100 ml samples.

sample	Comments
1	- To this 100 ml sample an additional 1 ml of NaOH was added and there was no change.
2	-To this sample 100 ml of water was added, at which time it was divided into samples 2a and 2b
2a	- This sample had nothing else done to it and was an example of what 2 would look like. The top layer was 30 ml and it was black. The bottom was 70 ml and it was brown.
2b	- This sample had 12 ml of NaOH added to it and after 24 hours the top 25 of a 115 ml was black. The bottom layer was brown.

NOTE: - Sample 1 had the darkest brown layer while 2b was the lightest.

- After adding 25 ml of water to sample 2, oil droplets started to form on the surface. This remained the same up to 100 ml was added.

TABLE C-4 FLUX CALC FOR OIL WATER MIXTURE

time (min)	temp °C	pressure inlet (psi)	pressure outlet (psi)	permeate rate mL/min	permeate rate USgal/day	flux USgal/ft2/day	oil conc	comments
								1:3 O/W pH 9.5
0							29%	HSC
10	41	70	50	60	22.8	194.7	30%	
30	39	70	50	40	15.2	129.8		
45	41	75	50	18	6.8	58.4	43%	
80	41	90	50	6	2.3	19.5	59%	
95	41	90	45	2	0.8	6.5	57%	
120	40	95	35	1.2	0.5	3.9	57%	
0							29%	HEM
10	41	70	50	46	17.5	149.2	30%	
30	39	70	50	32	12.2	103.8		
45	41	75	50	16	6.1	51.9	43%	
80	41	90	50	6	2.3	19.5	59%	
95	41	90	45	3	1.1	9.7	57%	
120	40	95	35	2	0.8	6.5	57%	
0							29%	HST
10	41	70	50	60	22.8	194.7	30%	
30	39	70	50	44	16.7	142.8		
45	41	75	50	18	6.8	58.4	43%	
80	41	90	50	5	1.9	16.2	59%	
95	41	90	45	2.5	1.0	8.1	57%	
120	40	95	35	2	0.8	6.5	57%	
0							29%	HST
10	41	70	50	58	22.1	188.2	30%	
30	39	70	50	40	15.2	129.8		
45	41	75	50	18	6.8	58.4	43%	
80	41	90	50	5	1.9	16.2	59%	
95	41	90	45	2.5	1.0	8.1	57%	
120	40	95	35	2	0.8	6.5	57%	
0							29%	HEM
10	41	70	50	50	19.0	162.2	30%	
30	39	70	50	40	15.2	129.8		
45	41	75	50	18	6.8	58.4	43%	
80	41	90	50	6	2.3	19.5	59%	
95	41	90	45	4	1.5	13.0	57%	
120	40	95	35	1.8	0.7	5.8	57%	
0							29%	HSC
10	41	70	50	62	23.6	201.2	30%	
30	39	70	50	46	17.5	149.2		
45	41	75	50	18	6.8	58.4	43%	
80	41	90	50	6	2.3	19.5	59%	
95	41	90	45	4	1.5	13.0	57%	
120	40	95	35	2	0.8	6.5	57%	

TABLE C-5 FLUX CALCULATION FOR THE STABLIZATION TEST AFTER DILUTING THE CONCENTRATE FROM FEB 1.
(AFTER DILUTION WITH 10 L OF TAP WATER)

TIME	TEMP	PRESSURE		PERMEATE RATE (ml/min)					
	°C	INLET	OUTLET	HSC	HEM	HST	HST	HEM	HSC
0	34	70	50	47	35	53	50	42	47
30	40	70	50	57	43	64	60	48	60
60	42	70	50	60	45	61	57	44	58
90	40	70	50	50	40	58	53	40	50
120	41	70	50	52	43	56	48	39	46
150	18	70	50	28	24	35	32	28	30
210	39	70	50	52	40	58	54	44	52
240	41	70	50	51	43	60	56	47	55

AVERAGE PERMEATE RATES

	permeate rate ml/min (OMIT DATA AT 18 °C)	permeate rate USgal/day	flux USgal/ft2/day
HSC	52.7	20.1	171.0
HEM	41.3	15.7	133.9
HST	58.6	22.3	190.0
HST	54.0	20.5	175.2
HEM	43.4	16.5	140.9
HSC	52.3	19.9	169.6

TABLE C-6 ORIGINAL PURE WATER RATE (STABLIZED)

(40° C/ 40° C) CLEANED PREVIOUSLY

	ml/min	permeate rate USgal/day	flux USgal/ft2/day	
HSC	99	37.7	321.2	
HEM	56	21.3	181.7	
HST	100	38.0	324.4	(not cleaned)
HST	89	33.9	288.7	
HEM	58	22.1	188.2	
HSC	95	36.1	308.2	

PURE WATER RATE FOLLOWING OIL/WATER DILUTION RUN

(FLUSHED WITH TAP WATER)

	ml/min	permeate rate USgal/day	flux USgal/ft2/day
HSC	18	6.1	51.9
HEM	15	5.7	48.7
HST	13	4.9	42.2
HST	12	4.6	38.9
HEM	17	6.5	55.2
HSC	20	7.6	64.9

TABLE C-7 CHARACTERISTICS OF THE DIALYSIS TEST PERMEATES

(mg/kg)	INITIAL WATER 1	WATER & NaOH 2	HSC 1 15 MIN 5	HEM 2 15 MIN 6	HST 3 15 MIN 7	HST 4 15 MIN 8	HEM 5 15 MIN 9	HSC 6 15 MIN 10	COMPOSITE 15 MIN 11	COMPOSITE 45 MIN 13	COMPOSITE 75 MIN 15	COMPOSITE 105 MIN 17	COMPOSITE 135 MIN 19	HSC 1,6 75 MIN 20
pH (20 °C)	-	-	-	-	-	-	-	-	-	-	-	-	-	-
Conductivity (μmho)	-	-	-	-	-	-	-	-	304	505	-	740	-	-
Oil & Grease	12	24	-	-	-	-	-	-	120	230	1100	930	880	860
Calcium	35	35	1.4	1.2	2.0	0.93	0.83	1.2	-	0.77	0.94	1.3	0.76	0.47
Magnesium	7.7	8.1	0.13	< 0.2	< 0.2	< 0.2	< 0.2	< 0.2	-	0.14	0.31	0.43	0.31	0.095
Sodium	13	240	70	85	90	88	80	68	-	130	180	200	110	72
Potassium	1.9	1.9	< 2	< 2	< 2	< 2	< 2	< 2	-	1.3	1.8	1.3	1.2	< 0.5
Aluminum	0.67	0.71	0.5	0.83	0.5	0.55	0.43	0.65	-	0.21	0.33	0.32	0.23	0.22
Barium	0.024	0.024	< 0.01	0.01	< 0.01	0.005	< 0.01	< 0.01	-	0.004	0.006	0.005	0.005	0.002
Beryllium	< 0.001	< 0.001	< 0.003	< 0.003	< 0.003	< 0.003	< 0.003	< 0.003	-	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001
Boron	< 0.1	< 0.1	< 0.3	< 0.3	< 0.3	< 0.3	< 0.3	< 0.3	-	< 0.1	0.2	< 0.1	< 0.1	< 0.1
Cadmium	< 0.002	< 0.002	< 0.006	< 0.006	< 0.006	< 0.006	< 0.006	< 0.006	-	< 0.002	< 0.002	< 0.002	< 0.002	< 0.002
Chromium	0.033	0.16	0.018	0.015	0.036	0.11	0.083	0.1	-	0.074	0.27	0.49	0.46	0.11
Cobalt	< 0.01	< 0.01	< 0.03	< 0.03	< 0.03	< 0.03	< 0.03	< 0.03	-	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01
Copper	0.072	0.086	0.033	0.043	0.023	< 0.03	0.053	0.015	-	0.016	0.028	0.024	0.029	< 0.006
Iron	0.32	1.5	0.08	0.43	0.33	0.55	0.50	0.45	-	0.1	0.11	0.049	0.66	0.2
Lead	< 0.04	< 0.04	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	-	< 0.04	< 0.04	< 0.04	< 0.04	< 0.04
Manganese	0.014	0.023	< 0.02	< 0.02	< 0.02	< 0.02	< 0.02	< 0.02	-	< 0.005	< 0.005	< 0.005	0.008	< 0.005
Molybdenum	< 0.02	< 0.02	< 0.06	< 0.06	< 0.06	< 0.06	< 0.06	< 0.06	-	< 0.02	0.043	0.06	0.05	< 0.02
Nickel	0.012	0.069	< 0.03	< 0.03	0.19	0.15	0.06	0.058	-	0.024	0.022	0.015	0.038	0.022
Phosphorus	< 0.1	< 0.1	0.3	0.35	0.43	0.4	0.25	< 0.3	-	1.4	3.3	4.3	2.8	1.1
Silicon	0.52	1	0.63	0.75	0.43	0.43	1.2	0.38	-	0.73	1.5	0.26	0.4	0.2
Silver	< 0.01	< 0.01	< 0.03	< 0.03	< 0.03	< 0.03	< 0.03	< 0.03	-	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01
Strontium	0.17	0.17	0.0075	0.0075	0.0075	0.005	0.005	0.005	-	0.003	0.006	0.007	0.005	0.002
Sulphur	9.4	9.8	22	22	55	30	25	16	-	45	58	61	36	22
Thallium	< 0.2	< 0.2	< 0.6	< 0.6	< 0.6	< 0.6	< 0.6	< 0.6	-	< 0.2	< 0.2	< 0.2	< 0.2	< 0.2
Titanium	0.033	0.055	0.14	0.12	0.12	0.14	0.11	0.13	-	0.033	0.034	0.047	0.035	0.048
Vanadium	< 0.01	< 0.01	< 0.03	< 0.03	0.14	0.028	0.03	< 0.03	-	0.031	0.034	0.031	0.013	< 0.01
Zinc	0.061	0.073	0.063	0.063	0.088	< 0.01	0.048	0.035	-	0.028	0.042	0.04	0.031	0.007
Zirconium	< 0.01	< 0.01	< 0.03	< 0.03	< 0.03	< 0.03	< 0.03	< 0.03	-	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01

TABLE C-8 CHARACTERIZATION OF THE DIALYSIS TEST CONCENTRATES

(mg/kg)	INITIAL 3	15 MIN 4	45 MIN 12	75 MIN 14	105 MIN 16	135 MIN 18
pH (20 °C)	9.23	9.77	9.77	9.34	9.42	9.64
Conductivity (µmho)	-	-	-	-	-	-
Oil & Grease	290000	300000	430000	590000	570000	570000
Calcium	8.8	10	22	19	56	56
Magnesium	2.6	2.1	5.5	5.5	12	13
Sodium	160	86	170	150	270	290
Potassium	< 20	< 20	< 20	< 20	< 20	< 20
Aluminum	1.8	1.2	1.2	1.8	5.3	5.6
Barium	< 0.05	< 0.05	0.063	0.054	0.26	0.23
Beryllium	< 0.02	< 0.02	0.035	0.021	0.062	0.067
Boron	< 0.5	< 0.5	< 0.5	< 0.5	< 5	< 5
Cadmium	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05
Chromium	< 0.1	0.26	0.84	0.55	2.5	2.3
Cobalt	< 0.3	< 0.3	< 0.3	< 0.3	< 0.3	< 0.3
Copper	< 0.2	< 0.2	0.36	0.32	1.4	1.3
Iron	0.48	0.35	1.7	1.3	6.5	4.0
Lead	< 1	< 1	< 1	< 1	< 1	< 1
Manganese	< 0.2	< 0.2	< 0.2	< 0.2	< 0.2	< 0.2
Molybdenum	0.61	< 0.5	1.5	1.1	3.5	3.7
Nickel	0.92	0.36	18	6.6	32	36
Phosphorus	< 3	< 3	< 3	< 3	6.6	5.7
Silicon	3.6	2.1	3.9	3.8	12	12
Silver	< 0.3	< 0.3	< 0.3	< 0.3	< 0.3	< 0.3
Strontium	0.12	0.1	0.25	0.19	0.55	0.54
Sulphur	980	440	6200	2800	17000	14000
Thallium	< 2	< 2	< 2	< 2	< 2	< 2
Titanium	0.36	< 0.3	0.95	0.64	4.4	4.4
Vanadium	4.2	1.1	46	26	95	100
Zinc	0.37	< 0.2	< 0.2	0.64	0.71	0.73
Zirconium	< 0.3	< 0.3	< 0.3	< 0.3	< 0.3	< 0.3

APPENDIX D

RAW RESULTS ALTERNATIVE STUDY

TABLE D-1 THE EFFECT OF pH ON THE THREE OIL FIELD SAMPLES

SAMPLE	COMMENTS
LLOYDMINSTER	-A LARGE AMOUNT OF NaOH WAS ADDED TO A MIXTURE CONTAINING 100 ml OF THIS BITUMEN AND 100 ml OF TAP WATER -THE INCREASE IN pH HAD NO EFFECT ON THE MIXTURE, THE WATER HAD A pH OF 14 -THE WATER WAS EXPECTED TO FIRST BE COLOURED AND THEN BECOME THINNNER AT WHICH TIME THE MIIXTURE WOULD TURN BROWN -FOLLOWING THIS, CONCENTRATED SULPHURIC ACID WAS ADDED TO THE MIXTURE BUT NO CHANGE OCCURED
COLD LAKE	-THIS SAMPLE HAD THE SAME RESULTS AS LLOYDMINSTER
PEACE RIVER	-A 100 ml OF SAMPLE WAS MIXED WITH 100 ml OF TAP WATER, 10 ml OF 0.1 N NaOH WAS ADDED AND THE WATER ON TOP STARTED TO TURN BROWN -WHEN 14.5 ml WAS ADDED, THE SOLUTION WAS VERY THIN AND TURNED BROWN -IT APPEARED THAT THE SOLUTION HAD REVERSED TO AN OIL IN WATER EMULSION -WHEN SULFURIC ACID WAS ADDDED TO THE SOLUTION NOTHING APPEARRED TO OCCUR.

TABLE D-2 VISCOSITY OF A WATER IN OIL (W/O) EMULSION AT VARIOUS TEMPERATURES

SAMPLE	TEMP. (deg. cel.)	SPINDLE MODEL AND NUMBER	SPEED	FACTOR	RANGE	READING	VISCOSITY (cps)	AVERAGE
Standard (55040cps @ 25 deg. cel.)	25	4	6	1000	100000	59.5	59500	
PEACE RIVER 1	23	4	60	100	10000	19.5	1950	
PEACE RIVER 1	25	4	60	100	10000	14.5	1450	
PEACE RIVER 1	40	4	60	100	10000	5.5	550	
PEACE RIVER 1	40	2	30	10	1000	54	540	
PEACE RIVER 1	60	2	30 60	10 5	1000 500	19 37.5	190 187.5	188.8
PEACE RIVER 1	80	2	60	5	500	16.5	82.5	

* Due to a low scale reading (< 10) of the previous measurement a larger spindle was chosen to increase acurracy
** Temperature fluctuation increased with an increase in temperature affecting reproducibility of certain results
NOTE : The container used to measure the viscosity of the sample was a 600 ml beaker
In all cases the spindle guard was used for the viscosity measurements.

TABLE D-3 VISCOSITY OF A WATER IN OIL (W/O) EMULSION AT VARIOUS TEMPERATURES

SAMPLE	TEMP. (deg. cel.)	SPINDLE MODEL AND NUMBER	SPEED	FACTOR	RANGE	READING	VISCOSITY (cps)	AVERAGE
Standard (55040cps @ 25 deg. cel.)	25	4	6	1000	100000	59.5	59500	
PEACE RIVER 2	25	4	6	1000	10000	42.5	42500	
PEACE RIVER 2	28	4	12	500	5000	88	44000	
PEACE RIVER 2	39	4	12	500	5000	45	22500	
PEACE RIVER 2	40	2	1.5	200	2000			
PEACE RIVER 2	60	2	3	100	1000	62.5	6250	188.8
PEACE RIVER 2	80	2	3 6	100 50	1000 500	22 40.5	2200 2025	2113

* Due to a low scale reading (< 10) of the previous measurement a larger spindle was chosen to increase acurracy
** Temperature fluctuation increased with an increase in temperature affecting reproducibility of certain results
NOTE : The container used to measure the viscosity of the sample was a 600 ml beaker
In all cases the spindle guard was used for the viscosity measurements.

TABLE D-4 VISCOSITY OF A WATER IN OIL (W/O) EMULSION AT VARIOUS TEMPERATURES

SAMPLE	TEMP. (deg. cel.)	SPINDLE MODEL AND NUMBER	SPEED	FACTOR	RANGE	READING	VISCOSITY (cps)	AVERAGE
Standard (55040cps @ 25 deg. cel.)	25	4	6	1000	100000	59.5	59500	
LLOYDMINSTER	28	4	60	100	10000	5	500	
LLOYDMINSTER	40	2	60	5	500	36	180	
LLOYDMINSTER	59	2	60	5	500	23	115	
LLOYDMINSTER	80	2	60	5	500	11	55	

* Due to a low scale reading (< 10) of the previous measurement a larger spindle was chosen to increase acurracy
** Temperature fluctuation increased with an increase in temperature affecting reproducibility of certain results
NOTE : The container used to measure the viscosity of the sample was a 600 ml beaker
In all cases the spindle guard was used for the viscosity measurements.

TABLE D-5 VISCOSITY OF A WATER IN OIL (W/O) EMULSION AT VARIOUS TEMPERATURES

SAMPLE	TEMP. (deg. cel.)	SPINDLE MODEL AND NUMBER	SPEED	FACTOR	RANGE	READING	VISCOSITY (cps)	AVERAGE
Standard (55040cps @ 25 deg. cel.)	25	4	6	1000	100000	59.5	59500	
COLD LAKE	26	4	12	500	50000	53	26500	
COLD LAKE	40	4	30	200	20000	56.5	11300	
COLD LAKE	40	2	1.5	200	20000	59.5	11900	
COLD LAKE	60	2	6	50	5000	62.5	3125	
COLD LAKE	80	2	12 30	25 10	2500 1000	39 96.5	975 965	188.8

* Due to a low scale reading (< 10) of the previous measurement a larger spindle was chosen to increase acurracy
** Temperature fluctuation increased with an increase in temperature affecting reproducibility of certain results
NOTE : The container used to measure the viscosity of the sample was a 600 ml beaker
In all cases the spindle guard was used for the viscosity measurements.

TABLE D-6 THE EFFECT OF A DILUENT ON BITUMEN VISCOSITY (AT 20 ° C)

SAMPLE	VISCOSITY CPS (25 ° C)	VISCOSITY (CPS) 4 : 1 (BITUMEN : DILUENT)	2 : 1 (BITUMEN : DILUENT)
ORIGINAL	1450	125	73.5
LLOYDMINSTER	500	90	43.5
COLD LAKE	26500	462	75
PEACE RIVER	42500	695	174

NOTE: THE DILUENT USED WAS VARSOL

TABLE D-7 OIL PERMEATION TESTS ON THE LLOYDMINSTER SAMPLE

time (min)	temp °C	pressure inlet (psi)	pressure outlet (psi)	flow l/min	permeate rate mL/min	permeate rate USgal/day	flux USgal/ft2/day	% water feed	% water perm	comments
0	40	60	40	6				4.4	0.18	-k750 membrane
15	40	60	45	5	5.00	1.9	28.28			
30	40	60	40	5	3.00	1.1	16.97			
60	41	60	30	6	1.00	0.4	5.66			
90	40	60	50	5	0.55	0.2	3.08			
120	41	60	40	5	0.32	0.1	1.80			
150	41	60	45	5	0.14	0.1	0.77			
180	41	70	40	5	0.18	0.1	1.03			
210	40	65	20	9	0.23	0.1	1.29			
240	41	80	25	9	0.45	0.2	2.57			
270	45	75	30	9	0.50	0.2	2.83			
300	42	75	20	10	0.45	0.2	2.57	4.7	0.18	

TABLE D-8 OIL PERMEATION TESTS ON THE COLD LAKE SAMPLE

time (min)	temp °C	pressure inlet (psi)	pressure outlet (psi)	flow l/min	permeate rate mL/min	permeate rate USgal/day	flux USgal/ft2/day	% water feed	% water perm	comments
0	43	80	50	0.1	0.05	0.02	0.28	19		-k750 membrane
30	43	95	38	0.25	0.05	0.02	0.28			
45	43	90	20	0.6	0.05	0.02	0.28			
60	42	95	20	0.6	0.04	0.02	0.23			
90	37	80	20	0.6	0.04	0.02	0.23			
120	37	80	20	0.4	0.04	0.02	0.23			
150	38	80	20	0.4	0.04	0.02	0.23			
180	38	85	20	0.4	0.04	0.02	0.23			
210	41	85	20	0.4	0.04	0.02	0.23			
240	42	90	20	0.4	0.04	0.02	0.23		<0.1	(≈0.3ml/hour)

TABLE D-9 OIL PERMEATION TESTS ON THE PEACE RIVER 2 SAMPLE

time (min)	temp °C	pressure inlet (psi)	pressure outlet (psi)	flow l/min	permeate rate mL/min	permeate rate USgal/day	flux USgal/ft2/day	% water feed	% water perm	comments
0	41	90	10	0.003	-			16		-k750 membrane
30	42	95	10	0.004	-					
45										-started collecting
60	43	90	12	0.005	-					permeate
120	46	90	13	0.007	-					
150	50	90	16	0.007	-					
180	50	95	22	0.007	-					
210	50	93	22	0.007	0.005	0.00	0.03		0.5	-finished collecting permeate (~0.3ml/hour)

TABLE D-10 **FLUX CALC FOR A 1 : 3 OIL WATER MIXTURE AT pH 9.5 (PEACE RIVER 2)**

time (min)	temp °C	pressure inlet (psi)	pressure outlet (psi)	permeate rate mL/min	permeate rate USgal/day	flux USgal/ft2/day	oil conc	comments
0	41	70	50	48	18.3	155.7	13%	HSC
30	42	70	50	52	19.8	168.7	-	
60	45	70	44	22	8.4	71.4	31%	
90	46	70	38	6	2.3	19.5	40%	
0	41	70	50	34	12.9	110.3	13%	HBM
30	42	70	50	35	13.3	113.6	-	
60	45	70	44	22	8.4	71.4	31%	
90	46	70	38	7	2.7	22.7	40%	
0	41	70	50	38	14.5	123.3	13%	HST
30	42	70	50	42	16.0	136.3	-	
60	45	70	44	30	11.4	97.3	31%	
90	46	70	38	7	2.7	22.7	40%	
0	41	70	50	34	12.9	110.3	13%	HST
30	42	70	50	34	12.9	110.3	-	
60	45	70	44	24	9.1	77.9	31%	
90	46	70	38	7	2.7	22.7	40%	
0	41	70	50	40	15.2	129.8	13%	HBM
30	42	70	50	44	16.7	142.8	-	
60	45	70	44	24	9.1	77.9	31%	
90	46	70	38	7	2.7	22.7	40%	
0	41	70	50	52	19.8	168.7	13%	HSC
30	42	70	50	59	22.4	191.4	-	
60	45	70	44	26	9.9	84.4	31%	
90	46	70	38	8	3.0	26.0	40%	

TABLE D-11 **FLUX CALCULATION FOR THE STABLIZATION TEST WITH THE PEACE RIVER 2 DIALYSIS CONCENTRATE**
(*-DILUTION WITH 10 L OF TAP WATER)

TIME	TEMP	PRESSURE		PERMEATE RATE (ml/min)					
	°C	INLET	OUTLET	HSC	HEM	HST	HST	HEM	HSC
0	40	60	50	45	31	36	27	35	49
30	40	60	50	46	30	36	26	34	48
60	40	60	50	46	30	34	25	36	48
90	40	60	50	44	30	34	24	36	48
120	40	60	50	43	43	30	22	34	50

AVERAGE PERMEATE RATES FOR STABLIZATION RUN WITH PEACE RIVER 2 BITUMEN

	permeate rate ml/min	permeate rate USgal/day	flux USgal/ft2/day
HSC	45.0	17.1	146.0
HEM	29.0	11.0	94.1
HST	35.0	13.3	113.6
HST	26.0	9.9	84.4
HEM	35.0	13.3	113.6
HSC	49.0	18.6	159.0

TABLE D-12 CHARACTERISTICS OF THE DIALYSIS TEST PERMEATES (PEACE RIVER 2)

Parameter (mg/l)	COMP PERM 15 MIN	HSC PERM 30 MIN	HEM PERM 30 MIN	HST PERM 30 MIN	HST PERM 30 MIN	HEM PERM 30 MIN	HSC PERM 30 MIN	COMP PERM 60 MIN	COMP PERM 75 MIN	COMP PERM 90 MIN	COMP PERM 90 MIN dup
pH (20 °C)	10.41	10.01	10.21	10.31	10.24	10.21	10.08	10.07	10	9.99	-
Conductivity (µmho)	0.505	0.699	0.877	0.711	0.849	0.868	0.767	1.341	1.386	1.422	-
H2O (%) -KFT	-	-	-	-	-	-	-	-	-	-	-
Oil & Grease	88	210	280	210	290	130	95	150	1700	800	-
Calcium	0.6	-	-	-	-	-	-	-	-	1.5	1.4
Magnesium	0.23	-	-	-	-	-	-	-	-	5.4	4.7
Sodium	120	-	-	-	-	-	-	-	-	380	340
Potassium	1.7	-	-	-	-	-	-	-	-	8.9	8.0
Aluminum	< 0.2	-	-	-	-	-	-	-	-	< 0.2	< 0.2
Barium	0.004	-	-	-	-	-	-	-	-	0.012	0.008
Beryllium	< 0.002	-	-	-	-	-	-	-	-	< 0.002	< 0.002
Boron	0.6	-	-	-	-	-	-	-	-	2.7	2.2
Cadmium	0.004	-	-	-	-	-	-	-	-	< 0.004	< 0.004
Chromium	< 0.01	-	-	-	-	-	-	-	-	0.15	0.13
Cobalt	< 0.02	-	-	-	-	-	-	-	-	< 0.02	< 0.02
Copper	< 0.02	-	-	-	-	-	-	-	-	0.022	0.024
Iron	0.03	-	-	-	-	-	-	-	-	0.27	0.25
Lead	< 0.08	-	-	-	-	-	-	-	-	< 0.08	< 0.08
Manganese	< 0.02	-	-	-	-	-	-	-	-	< 0.02	< 0.02
Molybdenum	< 0.04	-	-	-	-	-	-	-	-	0.17	0.15
Nickel	< 0.02	-	-	-	-	-	-	-	-	0.072	0.094
Phosphorus	1.2	-	-	-	-	-	-	-	-	21	19
Silicon	3.1	-	-	-	-	-	-	-	-	8.7	7.5
Silver	< 0.02	-	-	-	-	-	-	-	-	< 0.02	< 0.02
Strontium	0.002	-	-	-	-	-	-	-	-	0.012	0.01
Sulphur	10	-	-	-	-	-	-	-	-	59	54
Thallium	< 0.4	-	-	-	-	-	-	-	-	< 0.4	< 0.4
Titanium	0.048	-	-	-	-	-	-	-	-	0.052	0.05
Vanadium	< 0.02	-	-	-	-	-	-	-	-	0.052	0.054
Zinc	0.018	-	-	-	-	-	-	-	-	0.032	0.024
Zirconium	< 0.02	-	-	-	-	-	-	-	-	< 0.02	< 0.02

COMP PERM - COMPOSITE OF THE SIX MEMBRANE PERMEATES

TABLE D-13 CHARACTERISTICS OF THE DIALYSIS TEST CONCENTRATES (PEACE RIVER 2)

Parameter (mg/l)	INITIAL FEED	FEED 30 MIN	FEED 60 MIN	FEED 90 MIN
pH (20 °C)	10.11	9.94	9.56	9.26
Conductivity (µmho)	430	210 / 210	250	30
H2O (%) -KFT	71	-	-	36
Oil & Grease	133000	62000	310000	400000
Calcium	6.1	-	-	130
Magnesium	2.2	-	-	< 40
Sodium	150	-	-	450
Potassium	3.8	-	-	< 100
Aluminum	0.3	-	-	< 30
Barium	0.045	-	-	0.94
Beryllium	< 0.005	-	-	< 0.1
Boron	1.2	-	-	38
Cadmium	< 0.01	-	-	< 0.2
Chromium	0.04	-	-	< 5
Cobalt	< 0.05	-	-	< 5
Copper	0.05	-	-	< 5
Iron	0.69	-	-	21
Lead	< 0.2	-	-	< 10
Manganese	< 0.02	-	-	< 5
Molybdenum	0.34	-	-	< 5
Nickel	0.76	-	-	36
Phosphorus	4.0	-	-	< 50
Silicon	3.9	-	-	53
Silver	< 0.05	-	-	< 5
Strontium	0.07	-	-	1.6
Sulphur	450	-	-	13000
Thallium	< 1	-	-	< 15
Titanium	0.15	-	-	< 5
Vanadium	2.2	-	-	95
Zinc	0.17	-	-	< 5
Zirconium	< 0.05	-	-	< 5

COMP PERM - COMPOSITE OF THE SIX MEMBRANE PERMEATES